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*J. Charles*

**Philip Morris U.S.A.  
Research Center  
Richmond, Virginia**

**Period Covered**

**JULY 1 — 31, 1987**

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\*N/R = No Report

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PROJECT NUMBER: 2100  
PROJECT TITLE: New Product Innovations  
PROJECT LEADER: H. V. Lanzillotti  
PERIOD COVERED: July, 1987

I. LOW TAR/FULL FLAVOR

A. Objective: Employ novel cigarette construction and filter design concepts to develop a new low tar/full flavored cigarette product.

B. Status/Plans:

C Tube-in-Filter: 4mg. tube-in-tow prototype redesigned for reduced dilution. Non-standard tow obtained and Semiworks fabrication requested.

Laser chopper ordered to increase flexibility in hole spacing for laser perforated C.A. tubing. Tubing to be perforated at various hole sizes and spacing values for testing at FTR.

II. SMOKING OPTIONAL CIGARETTE

A. Objective: Develop a cigarette product which will provide a flavor delivery system for dry puffing, during periods when smoking is prohibited. This product will have a feature which permits normal smoking when desired.

B. Status/Plans: Latest prototypes screened subjectively. Flavored models to be panel tested by P.E.D. first week in August.

III. PACKAGING

A. Objective: To develop novel package designs.

B. Status/Plans:

Humidor Pack: Cigarette pack aging studies in progress to monitor moisture loss from moisture control devices containing potassium citrate/sugar solution. Subjective testing indicates that after one week under hot dry conditions, cigarettes packed with moisture control devices are subjectively preferred to control cigarettes stored under same conditions. Subjectives will continue to be monitored in order to assess magnitude of "freshness" advantage afforded by moisture control device.

IV. INSTRUMENTATION SERVICES

A. Objective: To evaluate commercially available testing equipment, develop test procedures, and explore new methods of cigarette testing.

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- B. Status/Plans: Purchase request submitted for integration of LaserMike circumference guage into Filtrona cigarette test station software by Filtrona. Instrument modifications to be completed by October, 1987.

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PROJECT NUMBER: 2105  
PROJECT TITLE: Filter Development  
PROJECT LEADER: W. A. Nichols  
PERIOD COVERED: July, 1987

#### I. FILTER MATERIALS, LTD.

- A. Objective: Develop polypropylene tow that is equivalent to cellulose acetate.
- B. Results: Increased variability seen in recent samples has been attributed to changes occurring in the crimper. A coating of material becomes deposited on the stuffer box and crimper rolls with time. The material appears to be titanium dioxide but analysis is being done to confirm the compound. This problem should be eliminated with new crimpers which are to be installed in two weeks.

Subjective screening of cigarettes produced at three tar levels continues. Analysis of data from the smoking should begin this week. Informal smokings have shown that the degree of difference between the CA models and FML models increases with increased tar delivery.

- C. Plans: FML Subjective Panel evaluations will be completed by September. Tow produced on the new crimpers will be evaluated to assess RTD and weight variation.

#### II. MENTHOL APPLICATION

- A. Objective: Provide support for the introduction of menthol on foil.
- B. Results: The foil mentholator in R&D was shipped to Louisville for installation. The Louisville mentholator will be returned to R&D for retrofitting of machine improvements.

Lepel Corp. has been contacted to supply induction heating equipment for rapid equilibration experimentation.

- C. Plans: Assist Manufacturing in the installation and start-up of the menthol applicators. Identify and evaluate induction heating effects on mentholated foil samples.

#### III. NEW FILTER CONCEPTS

- A. Objective: Develop unique products using novel filters.
- B. Results: Cigarettes produced with modified fluted plastic filters having a lower RTD (100mm), demonstrated the ability to shift and expand tar range. Tar delivery with unoccluded and occluded flutes was 4.9 mg and 17.5 mg, respectively.



A concept for eliminating the tube in the tube-in-tow filter was tested. A similar geometry, a cylindrical hole, was formed by bonding the filter fibers over a mandrel. Cigarettes smoked with the filter showed some change in RTD. Additional models will be produced to increase the effect.

- C. Plans: A PA-8 and Mk 9 are being obtained for further development of the modified plastic fluted filter.

The alternate tube-in-tow design will be tested for effects on tar delivery.

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PROJECT NUMBER: 2304  
PROJECT TITLE: Flavor Development - New Products  
PROJECT LEADER: Barbara G. Taylor  
PERIOD COVERED: July, 1987

I. STUDIO (J. Pflueger)

A. Objective: To develop subjectively acceptable cigarettes with reduced sidestream visibility with flavor characteristics similar to Marlboro Lights.

B. Status: Flavor-Low non-menthol - Work is continuing by evaluating several new aftercut systems on the present blend.

C. MgO/Trim - To enhance the flavor subjectives of the 17mm model with MgO, several new blends were developed. Subjective evaluation of these blends are underway.

C. Plans: Selection of A/C and casing for full flavor model.

3rd quarter, 1987

II. LOW DENSITY ROD (V. Willis)

A. Objective: To develop a continuous process for the production of reduced density cigarettes.

B. Status: Samples of the individual blend components were subjectively evaluated. From the evaluations, an all-lamina blend was developed by the Leaf Department. Evaluations are presently underway to select the best pectin to be used on future models.

III. FOAMED BINDER (V. Willis)

A. Objective: To develop a binder system that would improve coal strength, decrease loose ends, and allow for possible weight reduction without adversely affecting the taste of the cigarette.

B. Status: Cigarettes containing the OBI apple pectin from Switzerland were made in the Cambridge Light configuration and subjectively evaluated. Significant differences were seen between the control and test samples.

IV. PROJECT OLIVE (R. Mitchell)

A. Objective: To develop a casing and aftercut system that simulates the subjective characteristics of Caster.

B. Status: Results from the Japanese Product testing showed that both Olive I and Olive II were preferred over Caster by Caster smokers.

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V. MERIT/GALAXY - JAPAN (R. Mitchell)

- A. Objective: To develop three new products for the Japanese market  
- Merit-type, Kent-type and Tobacco/sweet-type.
- B. Status: All three models were completed and are being tested in Japan.

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PROJECT NUMBER: 2305  
PROJECT TITLE: Flavor Development-Brand Modification-Menthol/Distinctive  
PROJECT LEADER: H. M. Maxwell  
PERIOD COVERED: July, 1987

I. ALTERNATIVE FLAVORS:

A. Objective: To identify and qualify alternative sources for several flavoring materials.

B. Status:

C 1. MS

O POL testing on B&H 100 and Marlboro is scheduled.

2. Casing Materials

Cigarettes were made and accepted for POL testing.

C. Plans:

MS-PG POL Production

July/August, 1987

Casing POL's (Ship)

D July, 1987

II. FLAVOR CENTER LIAISON:

A. Objective: To provide R & D assistance regarding Flavor Center production and QA problems.

B. Status: Two Flavor Center issues regarding specification conformity and/or solubility considerations were addressed this month.

C. Plans:

Flavor Center Issues

A Ongoing

III. PROJECT BOND

A. Objective: To develop a product with similar smoking characteristics to Prince, manufactured in Sweden.

B. Status: Flavoring materials were supplied to PM Europe for prototype cigarette production.

C. Plans:

Cigarette Production  
(Mini-Primary)

July, 1987

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Cigarette Production  
(Factory)

August, 1987

#### IV. PROJECT MOOG

- A. Objective: To develop the expertise to produce cigarettes that are subjectively equivalent to Salem, Newport and Kool.
- B. Status: K-type cigarettes for POL testing were made and are acceptable.
- C. Plans:

Test K-type Cigarettes                      June, 1987

Make N-type POL cigarettes                  July, 1987

#### V. PROJECT FLEX

- A. Objective: For reasons of flexibility, economics and logistics, alternatives for fifteen PM flavors are being identified.
- B. Status: Seven alternative flavors have been identified. These represent all Flex flavors used in PM domestic brands. Semi-works cigarettes, Merit and Merit menthol, will be made for evaluation and conformation. A POL was requested, Merit versus Merit with Flex substituted flavors.
- C. Plans:

Identify Priority                              July, 1987  
Replacements

Identify 15 Alternatives                    September, 1987

#### VI. PROJECT GLEN:

- A. Objective:

To develop the capability to produce glycerin-free cigarettes that are subjectively equivalent to current products, for PM Germany.

- B. Status:

Glycerin-free Marlboro-type cigarettes were produced in R&D Semi-works. If these are found to be physically and subjectively acceptable, materials will be forwarded to PM Europe for glycerin-free cigarette production. If these are found to be acceptable, the program can be extended to other brands for Germany.

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**C. Plans:**

R&amp;D Prototypes

July, 1987

Evaluation of Prototypes

July/August, 1987

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PROJECT NUMBER: 2306  
PROJECT TITLE: Marlboro Standardization and International Support  
PROJECT LEADER: J. L. Spruill  
PERIOD COVERED: July, 1987

I. MARLBORO STANDARDIZATION

- A. Objective: Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status: Report was issued on NTM tests 1 and 2. Manufacturing test completed and report will be issued. June 15 production pick-up under evaluation and a July 23 production pick-up ordered.
- C. Plans: Complete June 15 production pick-up. Standard Run V tentatively scheduled for week of September 14, 1987.

II. DOMESTIC CIGARETTE DEVELOPMENT PANEL

- A. Objective: To provide subjective direction for programs within R & D and manufacturing locations.
- B. Status: Thirty-two panels completed during reporting period.

III. PROJECT GAS

- A. Objective: To develop an 85mm full-flavored prototype using blend components and flavor systems which will provide a natural blended product.
- B. Status: POL 3539 (Camel 85 vs. "All Natural" Prototype A) has been released for testing. Additional blends received for cigarette fabrication. Selection of new blend will be incorporated into low density rod program.

- C. Plans: Await results of POL 3539.

Evaluation of new blend models.

IV. FLAVOR RESOURCE DATA BANK

- A. Objective: Creation, customization and maintenance of flavor resource and data files for use by Flavor Development Division.
- B. Status: User training of divisional personnel in progress.

V. INTERNATIONAL SUPPORT

Japan

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- A. Objective: Develop flavor systems appropriate for use on a charcoal filtered cigarette.
- B. Status: Same.
- C. Plans: Same.

GENERAL

- A. Objective: Develop substitutions for specific casing components used internationally to enhance storage and ease in handling.
- B. Status: Control and test casing formulas being made.
- C. Plans: Cigarette making and evaluation.

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PROJECT NUMBER: 2307  
PROJECT TITLE: Flavor Investigation/Nonvolatile Flavor  
Investigation/Processed Tobacco  
PROJECT LEADER: W. R. Raymond  
PERIOD COVERED: July, 1987

## I. FLAVOR INVESTIGATION

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Analytical Support: Nine compounded flavors and fruit extract concentrates have been analyzed for water, solvents, sugars and volatile composition. Twenty three flavor house essential oil submissions have been characterized by GC and GC-MS. A complete volatile composition comparison by GC and GC-MS is in progress for seven folded mandarin oils (4X to 10X). Several flavor chemicals have been assayed for purity and impurity identification.

Three A/C flavors and components for Project "Trim" were characterized by GC. Anethole analyses were performed on control (MS) and test (MS-PG) A/C's for B&H and Marlboro POL testing. All were within specifications.

2. Project "ART": Two samples of extractor residues, one dissolved in ethanol and the other in PG, were fractionated by silica gel chromatography and analyzed by GC and GC-MS. The samples appeared to be mixtures of long-chain hydrocarbon waxes, fatty acids, fatty acid esters and tobacco flavor compounds (mega stigmatrienones and probably thumberganoids, etc.).
3. Tonka Bean: Subfractionation and analytical characterization of major fractions continued. Monadic subjective testing on uncased Marlboro Lights blanks is in progress to select fractions for further evaluation and establish application windows.
4. Nontobacco Materials: Several samples of packaging materials, including new graphics samples of B&H labels and "Blue" labels from "Alford", were analyzed by headspace GC for residual ink solvents. "Golden Belt" foil, suspected of having an off-odor, was compared by GC to an acceptable control and found to be analytically no different.
5. Applied Flavor Investigation: A study was begun of the adsorptive properties of Ambersorb XW-340 and XE-348 using headspace-GC techniques. The objective is to test feasibility of flavor release systems based on these adsorbants.

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## II. NONVOLATILE FLAVOR INVESTIGATION

A. Objective: To investigate tobacco-natural, tobacco-identical and other nonvolatile fractions and constituents for potential application as cigarette flavor additives.

B. Results:

Initial size-exclusion (S.E.) experiments resulted in separation of Bright tobacco extract into three major fractions. Total nicotine content in the three fractions was determined by GC to be <2% of that present in the starting extract indicating adsorptive retardation by the S.E. medium. Fractionation will continue. Fractions will be assayed for glycoside content and evaluated subjectively.

## III. PROCESSED TOBACCO

A. Objective: To develop basic and applied knowledge for the purpose of improvement or selective modification of subjective properties of processed tobaccos.

B. Results:

1. POL's of humectant rearrangement in Marlboro, Marlboro Lights and B&H Menthol have been subjectively screened and released.
2. Repeat POL's of SHIS versus ES in Marlboro have been extended to include a casing modification. Awaiting stem from LVL. SHCRS to be evaluated for substitution in PML.
3. POL 3536 (Park 500 RL evaporator test) was released. A remake of POL 3535 cigarettes has been requested due to unacceptable subjectives in both control and test.
4. Internal subjective results showed differences for preservative added to flavor solution in RCB. Further testing recommended to determine potential interactions.
4. Cigarettes for POL's 3548 and 3549 (dry flavor replacements in RLB and RCB) scheduled to be made in early August.
5. POL's approved to evaluate modified RLB at various levels of RCB substitution and as total replacement for recon. Small scale preliminary testing in progress to evaluate effectiveness of substitution levels.
6. Subjective testing conducted of Bremen-4 stem, "as is" and "post washed," directly replacing ES in Marlboro and replacing Bright stem in RL's. Both Bremen-4 variables replacing ES in Marlboro yielded negative subjectives compared to the control, the "as is" contributing a bitter, metallic off-taste and the latter similar character with increased harshness and

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hotness. RLB with post-washed Bremen-4 stem was characterized as more hot, peppery and bitter/green than the "as is" variable both at the 24% level in Marlboro and in 100% RLB cigarettes. Post-washed stem substitution in RLTC, however, was characterized as less hot, peppery, bitter/green and mouthcoating than the "as is" variable at 24% in Marlboro. Further testing in progress to confirm above results as well as to evaluate variables at normal sheet levels in Marlboro.

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PROJECT NUMBER: 4009  
PROJECT TITLE: Development Smoke Studies  
PROJECT LEADER: B. L. Goodman  
PERIOD COVERED: July, 1987

I. PROJECT STUDIO (John Hearn)

- A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream visibility.
- B. Status: The OC Panel retest of the Marlboro Lights type model was still rated as less acceptable than the Marlboro Lights control, although none of the attributes were significantly different. Another aftercut system has been selected for the Flavor Low prototype for extended testing on the OC Panel along with the Marlboro Lights control. Evaluation of cigarettes with wrappers containing either citrate or acetate burn additives has determined that no significant analytical differences or subjective preferences were found between them. Another set of bobbins has been requested from Ecusta for a final subjective comparison. Ultra Low tar prototypes have been evaluated and an aftercut has been chosen for the next series of cigarettes in the 4-5 mg tar range.

Full Flavor versions with several construction variations have been made. Subjective evaluations will begin when the analytical data is completed.

Blend modifications suggested by the Leaf Department have been evaluated in the Ultra Slim construction by the Studio Panel. New models have been requested with blend changes and filtration modifications to further improve the subjective characteristics. Models with two levels of menthol will also be made. The best regular and menthol model will be tested by PED with the TRIM candidate without  $Mg(OH)_2$  paper.

Tests are in progress to determine the machineability of the standard  $Mg(OH)_2$  wrapper at production speeds. Currently, testing of different seam adhesives is being done with rods of 24.8mm circumference, but the ultimate goal is to qualify the wrapper for use in Project TRIM models.

Cigarettes were evaluated with different levels and types of burn additive on a 40%  $Mg(OH)_2$  wrapper. The cigarettes were very slow burning and did not have an acceptable taste with the blend (#102) that was used. Additional models with high levels of expanded tobaccos have been requested.

- C. Plans: Evaluate the latest series of Ultra Slim models and recommend candidates for consumer testing by PED in mid-August.

Continue with trials to evaluate the suitability of the  $Mg(OH)_2$  wrapper for use at production speeds and transfer the information to the appropriate personnel.

Subjectively evaluate the Full Flavor and new Ultra Low tar models.

II. TIPPING PAPER (R. Arthur)

- A. Objective: To evaluate alternate cork tipping papers.
- B. Results: Three samples of iron oxide base tippings were submitted by Ecusta and one by Kimberly-Clark. The tippings were in response to a request to meet German regulations for tipping colors. The tippings were laser perforated and cigarettes were made from the bobbins along with a control Ecusta 701 bobbin, the current production tipping. Filter flare-up testing gave unacceptable results ranging from 2% to 20% for the new tippings compared to the control with 0% flare-up. The results have been discussed with the vendors, and no further testing will be done. Modified samples are expected at a later date.

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PROJECT NUMBER: 2304, 2305, 4015  
PROJECT TITLE: New Product Development  
PROJECT LEADER: B. G. Taylor, H. Maxwell, and W. G. Houck  
PERIOD COVERED: July, 1987

I. MARLBORO/MARLBORO LIGHTS MENTHOL

A. Objective: (1) Develop Marlboro Menthol to appeal more to Salem smokers. (2) Develop a Marlboro Lights Menthol brand extension to appeal to Salem Lights smokers.

C B. Status: St. Louis Test Market Start-up, August 3, 1987. 85 and 100 mm products in place. 85 mm Marlboro Lights vs. Salem Lights POL released. National introduction to possibly include 80 mm (FF) and 83 mm (LTS) box packings.

C. Plans:

St. Louis Test Market	8/3/87
Complete POL's 2139/40	9/87
Develop specifications for box product	8/87
Monitor Test Markets	On-going

II. PM BLUES

A. Objective: To develop both full (16 mg) and light (12 mg) versions of distinctively flavored menthol candidates to appeal to 18-25 year old smokers.

B. Status: Atlanta Test Market cancelled. Blues LTS POL making in progress. Blue II (version I) scheduled for non menthol mapping mail-out panel.

C. Plans:

Complete LTS 85/100 POL Testing	9/87
Blue II Mapping	10/87
Monitor Cleveland Test Market	On-going

III. FRESH

A. Objective: To develop 12 mg free-standing menthol candidates with special packaging for "freshness" perception. Target market: Salem Lights smokers.

B. Status: Ad/Pack shipment to HTI 6/26/87. POL making for 100 mm model vs. Salem LTS SP and Salem LTS FTB (8 sided test market box) in progress.

C. Plans:

Complete POL Testing	10/87
Test Market	1988

IV. MARLBORO ULTRA LIGHTS

A. Objective: To develop 85/100 mm low (8 mg) and Ultra Low' (6 mg) candidates for Marlboro line extensions.

B. Status: POL Testing of 85/100 -- 6/8 mg prototypes complete. Both blend configurations appear to be within the range of acceptable candidates.

C. Plans:

Design Optimization (100 mm)	8/87
Additional POL Testing (If warranted)	10/87
Ad/Pack Testing	To be scheduled
Test market	1st Qtr., 88

V. PROJECT EXTRA

A. Objective: The development of 4-8 mg product candidates that have the subject taste of product with twice the tar.

B. Status: Mapping studies in progress. Paper/ca candidates under internal evaluation for selection of POL candidate(s). Flavor development with CR chemicals continue.

C. Plans:

Sensory mapping	On-going
Paper/ca POLs	10/87
Flavor development/screening	On-going

VI. PROJECT TRIM (Ultra Slim)

A. Objective: To develop 17 mm circumference/100 mm Ultra Slim product candidates.

B. Status: Mall testing of TRIM vs. Capri, B&H Lights, and Va. Slims --- all in regular and menthol --- in progress. Blend evaluation for  $Mg(OH)_2$  candidates in progress. Initial adhesive runability trials completed 7/30. Blend samples for FTR scheduled for the weeks of 8/3 and 8/10/87. Set-up filters for FTR to be mailed

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week of 8/3/87. B&H, Va. Slims, and Merit families under consideration for this product.

C. Plans:

Complete Mall Testing	8/87
Samples to FTR	8/87
Design Optimization	On-going
Preliminary Specification (Mg(OH) <sub>2</sub> and regular)	8/87
Test Market Start-up	10/87

VII. PROJECT ART

A. Objective: To investigate methods to decrease the nicotine content in tobacco. To develop full-flavored, ultra low, and light products using the low nicotine tobacco products.

B. Status: Flavor development continues on the 6, 11, and 16 mg tar models (menthol and non-menthol) to increase the acceptability of the extracted samples.

Components that were identified as having been removed, are presently being evaluated on the 6 mg AB-Art model. Four components thus far are showing some promise - damascone, geranyl acetone, guaiacol, and 2-acetyl pyridine.

RL-TC and RL-150B produced with the stems used in Bremen 4 runs have been subjectively evaluated. The RL-TC was more acceptable than the RL-150B in handmade samples. Subjective evaluations were also done on models where direct inclusion of the stems in the blend was done. The "as is" stem inclusion model was significantly better than the "post washed" stems inclusion model. Samples were also made utilizing the DIET expansion process for the stems. Subjectively, this model was not as good as the "as is" or "post-washed" direct inclusion models.

From evaluations of tower extracted washed burley stems, a blend (using Bright and Burley tobaccos only) was developed by the Leaf Department and submitted to the tower for extraction.

The Extended Smoking testing of Brem-4 and Doral has been completed. The ART model was significantly lower in acceptability

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than the Doral initially and at the end of five packs. The ART model's ratings dropped from the first to the last cigarette of the first pack where the Doral's ratings increased. The Doral ratings dropped from the first cigarette of the last pack to the last cigarette of the last pack where the ART model did not. Testing is being scheduled for the extended smoking of Carlton and Brem-4 model.

Sidestream visibility results for samples constructed at slim (23.0 mm) and ultra slim (17.0 mm) circumferences indicated a reduction of seventy-four percent for model X6D7BGH and seventy percent for model X6D7BGI. Both models were produced using  $\text{Mg(OH)}_2$  cigarette wrapper (P4LP).

C. Plans:

Continue flavor work on the 3, 6, 11 and 16 mg models	
Begin development of menthol ART models	3rd quarter
Complete OV study	July, 1987
Continue coordination of cigarette model production	On-going
Review program	3rd quarter, 1987
Pilot Plant start-up	Oct., 1987

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PROJECT NUMBER: 4022  
PROJECT TITLE: International Product Development  
PROJECT LEADER: R. E. Tinker, L. Mims Evans, C. B. Altizer  
PERIOD COVERED: July, 1987

I. PROJECT M-86 (PANAMA)

- A. Objective: Develop a local menthol product to compete with Kool.
- B. Results: Top line results of single pack test (N=150) vs. Kool indicate a slight preference for the test cigarette (No. 845). Complete SPT Report will be available August 10.
- C. Plans: An extended consumer test may be conducted to verify the single pack test results.

II. CHESTERFIELD MENTHOL (COSTA RICA)

- A. Objective: Relaunch Chesterfield Menthol with new blend and casing system.
- B. Results: Blends, casings and flavors have been submitted to Costa Rica for factory trials.
- C. Plans: Prepare and evaluate prototypes in Costa Rica in August 1987.

III. PROJECT COUNTRY (PHILIPPINES) (L. M. Evans)

- A. Objective: Bring the smoking characteristics of Marlboro closer to the characteristics of the Richmond - produced Marlboro.
- B. Results: The project flavor system has been finalized and the prototype will be produced for consumer testing as soon as the reconstituted tobacco is available for the blend. The recon plant is currently on strike.
- C. Plans: The new prototype will be consumer tested as soon as reconstituted tobacco is available.

IV. METRO (PHILIPPINES)

- A. Objective: Develop a low cost mentholated product (Monterey) for the middle or low price category.

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B. Results: The product has been produced for possible market introduction in September. Samples have been sent to Richmond for evaluation.

C. Plans: No additional work is required at this time.

V. MARLBORO (MALAYSIA)

A. Objective: Increase the amount of Malaysian tobacco in the blend.

B. Results: Two prototypes (control and test) were produced in Malaysia for consumer testing. The Malaysian tobacco in the blend was increased from 24.6 to 35.0 percent. Based on the objective and subjective evaluation of the cigarettes, they were not different.

C. Plans: The last shipment of strips to Malaysia was BBS #7, which is for the increased level of Malaysian tobacco.

VI. PROJECT BALTHAZAR (JAPAN) (C. B. Altizer)

A. Objective: Develop a Marlboro Lights with a charcoal filter and 50% Japanese tobacco to be manufactured by JTI.

B. Results: Subjectively and analytically acceptable prototype with a 15 CA/10 charcoal filter. Product team has presented JTI with preliminary specifications. Analytically acceptable prototypes have been constructed using four new blends.

C. Plans: Subjective acceptability of new blends is pending; prototypes using JTI cigarette and tipping papers will be constructed in early August. Development of a sixteen milligram balthazar has been initiated; prototypes to be constructed early August.

VII. PROJECT STARSHIP (JAPAN)

A. Objective: Develop a 12 mg. charcoal filter product which in conjunction with a young American image will compete successfully with the latest US imports.

B. Results: Test flavors have been made and requests submitted to Semiworks, using Chesterfield improved blend.

C. Plans: First series of prototypes to be available in August.

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VIII. PROJECT MERIT/GALAXY (JAPAN)

- A. Objective: Develop a low delivery (7 mg.) product with full flavor response.
- B. Results: Four seven milligrams prototypes have been developed and sent to Japan for product testing on Danchi Panel:
- 1) USA Merit Blend with Mount type filter.
  - 2) Mount Blend/Merit AC with Mount type filter.
  - 3) Mount Blend/Kent direction with Mount type filter
  - 4) Mount Blend/Distinctive flavor with Mount type filter
- C. Plans: Three PMI product tests are scheduled to be completed the week of August 3, 1987.
- 1) Mild Seven Lts. vs. Merit I (7 mg. Mount Blend/Kent Distinction with flush-fluted filter)
  - 2) Mild Seven Lts. vs. Merit II (7 mg. Mount Blend/Kent Direction with Mount type filter)
  - 3) Mild Seven Lts. vs. Merit III (7 mg. Mount Blend & Flavors with flush-fluted filter)

IX. PROJECT OLIVE (JAPAN)

- A. Objective: Develop an ultra low tar cigarette to compete with Caster K.S.
- B. Results: Danchi panel testing complete. Oliver I (8 mg.) was preferred over Olive II (6 mg.). PMI topline results complete; Olive I and Olive II both preferred over Caster King Size.
- C. Plans: Product Development complete, pending launch decision.

X. LARK IMPROVEMENT (JAPAN)

- A. Objective: Optimize Lark family in order to achieve higher consumer acceptance in blend product testing.
- B. Results: Lark Milds prototypes and Lark full flavor prototypes to be available in early August.
- C. Plans: Test both prototypes on Danchi Panel.

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XI. MOUNT 11MG (JAPAN)

- A. Objective: Redesign current PM Lights using Mount blend (11 mg tar)
- B. Results: Prototypes produced and shipped to Japan for Danchi and PMI product testing.
- C. Plans: Awaiting test results.

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PROJECT NUMBER: 5001  
PROJECT TITLE: Packaging Studies  
PROJECT LEADER: H. R. Dunaway  
PERIOD COVERED: July, 1987

#### PACKAGING STUDIES

A. Objective: Assist New Products Directorate in evaluating new packaging concepts and products, and provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing, and Quality Assurance.

B. Status/Plans:

1. Project Fresh: The Ad/Pack aging study has been completed, and confirms earlier test findings that the proposed packaging configuration improves moisture retention over conventional packaging. Since the Ad/Pack test was postponed, this study will be repeated when quality problems have been resolved and product is remade.

2. Project Fox: Boxes made of Philips BMN-55500 high density polyethylene with green pigment were jarred with cigarettes, aged, and the cigarettes subjectively evaluated. These cigarettes were found to be comparable in taste and odor to the controls jarred with a standard white SBS FTB. This material, however, does not provide the lid hinge with acceptable strength.

Samples made of a polyethylene/polypropylene mixture, for added strength, were received and testing is in progress.

3. B&H Graphics Change: A group of 4 SP labels and 2 FT boxes, scheduled for packing this month, were sampled upon arrival at our warehouse for GC/Headspace and subjective smoking analyses. Although residual solvent content was marginally high in some of the samples, subjective smoking results were favorable in all cases.

4. Polyester Tear Tape: Two gauges of clear polyester tear tape from Kable Tapes, Ltd. were compared to standard gold polypropylene tear tape on Cambridge Lts. 100 SP. Samples were placed under Lab, Jungle, Desert, and Cold conditions for a period of six weeks and evaluated for function and appearance. Each of the samples functioned properly under all conditions tested, but although none of the tapes broke during normal pack opening, it was noted that the test tapes were not as strong as the control and could be readily broken by hand.

5. Cigarette Case Overwrap: A study is underway to compare OV changes in full cases of SP and FTB export product to assess the effect of overwrapping the case versus overwrapping the individual cartons. Foil and polypropylene carton overwraps are included.

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PROJECT NUMBER: 0400  
PROJECT TITLE: Low Density Rod Development  
PROJECT LEADER: R. S. Mullins  
PERIOD COVERED: July 1987

#### LOW DENSITY ROD

- A. Objective: Develop a continuous process for the production of reduced density cigarettes.
- B. Results: Physical test results from the alternate pectin test indicated that the Bulmer pectin produces a firmer cigarette than either the Hercules L200, the Hercules X6950, or the degraded Hercules L200. The 57 mm cigarette rods produced with the Bulmer pectin had an equilibrated firmness of 3.1 mm at a tobacco weight of 515 mg while the firmness of the next closest candidates, the Hercules L200 and X6950, was approximately 3.7 mm at the same tobacco weight. However, since the L200 was subjectively preferred over the Bulmer, the L200 will be used for future samples produced for Flavor Development.

The binder application process was shut down during July to allow the process to be relocated and upgraded. One of the two binder application systems has now been returned to operation, allowing the production of samples for Flavor Development and New Products to resume.

The ecreteur on the low density cigarette maker was modified by removing the ecreteur disks and replacing the wiper disk with a spare ecreteur disk from the Hauni Pilot Maker. With this modification, a significant ecreteur return rate was obtained (20% - 25%) and the tobacco rod appeared more uniform. Based on a limited number of unequilibrated, hand weighed samples, this modification reduced the cigarette weight standard deviation from 35 mg to 24 mg. To confirm these preliminary results, additional cigarettes have been submitted for equilibration and weight selection.

Design of an improved microwave drying cavity has been completed. Fabrication of the cavity is scheduled to be completed by September 1.

- C. Plans: Start up the second binder application system. Complete fabrication of the improved microwave drying cavity. Produce additional samples for the Low Tar/High Flavor program from the following blends: Bremen IV without AB, cased Bremen IV with AB, all lamina, All-Natural, and the all lamina with MAP. Investigate the difference in binding performance between the Bulmer and Hercules L200 pectins. Identify the additional process development needed and the resources required to support product development (including POL's) and commercialization of the process.

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PROJECT NUMBER: 1005  
PROJECT TITLE: Semiworks Operations Support  
PROJECT LEADER: E. G. Craze  
PERIOD COVERED: July, 1987

I. ENGINEERING DEVELOPMENT (D. E. Albertson)

- A. Objective: Provide engineering and technical support to improve the performance and efficiency of the Semiworks operation.

B. Results:

Tobacco Lot Analysis (Skidmore/Nguyen) - Modifications to the Labcutter that made the unit self feeding were completed in July. This results in a significant improvement in the repeatability of the TLA primary process. A series of tests is being conducted to determine the optimum cutting conditions.

A test program is underway to determine the standard deviations for sample dependent, non-controllable, variables such as sieve sizes and CV for filler processed in the TLA primary. This information will be used to define the variations in parameters from a given tobacco grade lot.

ES Feeder Performance (Nguyen) - The Semiworks ES feeder has been redesigned to improve the performance of the ES addition system. The new feeder is being fabricated by Rayco and is scheduled to be installed beginning August 12, 1987.

Make/Pack (Albertson) - Installation of the first of four new MAX-S tippers has been initiated. Each unit is expected to take approximately three weeks to install with work continuing sequentially until all units have been installed.

The new high speed ring tipper was received from Hauni of Richmond and successfully run on a POL test. This unit is expected to improve product quality while reducing sample processing time.

- C. Plans: Develop a primary processing system for the Tobacco Lot Analysis Program and establish firm acceptance criteria for the TLA process.

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## II. PROCESS CONTROL (D. A. Phan)

- A. Objective: Evaluate and revise the Semiworks process control and data acquisition systems to improve processing performance and production quality.

B. Results:

Flavor Cylinder Priming (Oliver) - An automatic priming system has been implemented on the bright casing cylinder for improved flavor application during start-up. This completes the installation of automatic priming systems on all flavor cylinders in the Semiworks.

Flavor System Upgrade (Phan) - The design of the improved flavor system for the burley spray cylinder is complete. A new 100-gallon burley storage tank has been received and installation is planned for the week of August 24.

Optical Detection System (Oliver) - The modicon software has been revised to accomodate provisions for routing tobacco from both the 3,000 and 6,000-lb lines through the detection system for removal of foreign material in the preblend area. Construction work will begin August 14 and is scheduled to be complete by August 22.

Vacuum Conditioner Upgrade (Oliver/Sims) - work is continuing to provide identification tags on all of the equipment associated with the vacuum conditioning system to enhance operation and maintenance. In addition, existing drawings and control loops are being verified to assure the system is performing as designed.

Instrument Evaluations (Sims/Coleman) - Testing of the Bearingless volumetric flowmeter for use in applying aftercut flavors has been completed. The data are being evaluated and a final report is being prepared.

Testing of the Computrac moisture analyzer for use in determining moisture levels for filler release in the Semiworks primary is progressing. Preliminary results look promising and the evaluation is expected to be completed by October, 1987.

- C. Plans: Complete the checkout of the revised burley spray flavor system and the electrical interlocks for the optical detection system. Continue the evaluation of new instrumentation and conduct routine QA functions.

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PROJECT NUMBER: 1307  
PROJECT TITLE: Reconstituted Tobacco Development  
PROJECT LEADER: R. G. Uhl  
PERIOD COVERED: July, 1987

I. IMPROVED SHEET PROPERTIES

A. Objective: Improve the physical characteristics and blend performance of reconstituted sheet materials.

B. Results:

- C
1. ART Project - During the July shutdown period, the BL Plant produced an additional 55M lbs of washed stem for ART pilot plant use. Coupled with June production, this should meet projected needs through year end. These are mostly bright stems (1, 2, 3 or 4 fresh water washes), plus some 4X washed burley stems. Larger quantities of BL Plant 1X washed burley stems are available from production inventory.  
  
R&D participated in off-site Engineering trials to evaluate the Rietz V-Press for use in stem washing. The unit accepted any tobacco slurry fed to it. Attrition was minimal, even with shredded stems. Press cake OV was 64-72%. However, the press seals leak considerable amounts of liquor, and the press liquors have a high-fines content.
  2. Humectant Rearrangement - The first three humectant rearrangement POL tests (test blends made with reduced humectant level sheets) are closed out and data is under analysis. Cigarettes for the three replicate POL tests have been completed in Semiworks.
  3. Alternate Evaporator Technology - POL cigarettes to evaluate the Engineering Wiegand thin-film evaporator test at Park 500 have been sent to the panelists. Cigarettes for the replicate POL are being screened for mailout approval.
  4. TTG Support - BL Plant RCB made with Malaysian feedstocks survived much better than control RCB, and gave sieve profiles (exit cutter, dryer, maker garniture) typical of RL. The test containing humectants did particularly well, and was long and stringy enough to cause problems at the maker (100% sheet). This sheet had a higher tensile than the control (0.9 vs 0.7) at a lower sheet weight (10.0 vs 11.1). The test sheets had a higher stem content (65%); the stems were unwashed; the stems were ground and fed in through the CT system. Cigarettes (100% only) were produced on small scale for subjective screening.
  5. Sidestream Reduction - RCB handsheets were made with up to 30% magnesium hydroxide to evaluate filler incorporation for sidestream reduction mechanism studies. At 10% and above, the sheets became progressively lighter in color and developed belt-release properties, but the slurries gelled quickly, the

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sheets became progressively brittle and piece size (after shredding) was drastically reduced. Some handmade cigarettes were produced from all but the 30% sample.

C. Plans:

1. Expedite subjective evaluation of pilot RL containing Bremen-4 ART stems.
2. Evaluate Quaker Yankee dryer release agents.
3. Produce PGG-free sheets for the Humectant Replacement Group.

II. SUBJECTIVE MODIFICATION OF RL

A. Objective: Improve or modify the subjective character of RL.

B. Results:

1. The production of POL cigarettes to evaluate RCB and RL-150B made with dry flavor replacements has been scheduled for August in Semiworks.
2. POL cigarettes to evaluate Park 500 Modified 150B (5% DAP) replacing standard 150B plus 3% and all of the RCB, and as a total replacement for all sheet, have been scheduled for August in Semiworks.
3. Efforts to eliminate the magnesium/calcium phosphate precipitate from Modified 150B size continued. SEL was treated with DAP (and adjusted to several pH levels with ammonium hydroxide) to pre-form the precipitate and allow its removal with the normal SEL centrifuge sludge. Alkaloid-like off-odors were noted during liquor processing and sheet drying, and there was a considerable falloff in evaporation rate. Analytical and subjective results are pending.
4. Magnesium salt removal from DN-CEL has been demonstrated, but this would require an additional 2-stage centrifuging step at Park 500. Current efforts are attempting treatment of undenitrated CEL to remove magnesium along with the potassium nitrate co-product.
5. Laboratory investigation of other sequestering agents indicates that tripolyphosphates sequester magnesium and calcium in a non-precipitate form, eliminating the need for removal. Larger quantities have been ordered for pilot scale trials.

C. Plans:

Continue pilot and laboratory programs to eliminate magnesium phosphate precipitate.

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PROJECT NUMBER: 1503  
PROJECT TITLE: Modified Smoking Materials  
PROJECT LEADER: J. G. Nepomuceno  
PERIOD COVERED: July, 1987

#### FOAMED FILLER BINDER

- A. **Objective:** To develop a process for applying a subjectively acceptable foamed binder to the tobacco filler during making to improve cigarette coal strength, reduce loose ends, increase firmness and allow for weight reductions.
- B. **Results:** A series of degraded pectin binders with average molecular weights ranging from ~2000 - 8000 MW was evaluated on Cambridge Lights cigarettes. These pectin binders were prepared in-house using the 5 gallon lab vessel. Included in these evaluations were pectin binders received from Grindsted (a commercial pectin supplier). The pectin binders with molecular weights from 3000 to 5000 gave promising results. The pectin binder with an average molecular weight of 4000 applied to the cigarette at a 0.3% addition rate using a 35% binder solution showed the most potential for firmness improvement, yielding a 0.25mm firmness improvement at equal OV's (cigarettes equilibrated in a 56% RH cabinet). At this addition rate, the pack OV increased by ~0.4% due to the binder application, yielding a pack firmness (unequilibrated) improvement of 0.05 units. Follow-up runs are in progress where the OV of the tobacco was adjusted to compensate for the 0.4% increase in moisture. This same binder solution was also applied to the cigarette at a 1.0% addition rate yielding a firmness improvement of approximately 0.5mm units at equal OV's. At this 1% addition rate, the pack moisture was increased by ~1.2% yielding a pack firmness loss of 0.1 units. Additional runs are also in progress for this add-on rate where the tobacco OV was adjusted to compensate for the 1.2% increase in pack moisture.

Binder solutions with glycerin added to the formulation were also prepared in effort to increase the volume of binder added at the low pectin addition rates (thus increasing the number of bound shreds) without significantly increasing the amount of moisture added. A 0.2 g/cc foam density was achieved with the addition of 1% foaming agent (yucca concentrate). This formulation will be applied to the cigarette and evaluated for firmness improvement.

Tobacco filler is being prepared where the degraded pectin is added to the tobacco via the total blend casing. This technique is being explored as a method of facilitating blend and flavor development for the foam bound cigarettes.

The pilot plant process for the continuous degradation of pectin binders was installed and started up this month. Start up runs have gone smoothly to date. Problems with the fine tuning of the temperature controller were identified and are being rectified. Additional trials are currently in progress in an effort to establish process operating conditions.

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- C. Plans: Complete evaluation of the 3000 to 5000 MW pectin binders at the 0.3 and 1.0% add-on rates using moisture adjusted feed tobacco. Provide tobacco with pectin binder applied through the total blend casing to product development for blend and flavor development. Complete start-up trials on the continuous degradation process and begin producing thermally degraded pectin for evaluation on foam bound rods.

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PROJECT NUMBER: 1801  
PROJECT TITLE: Expanded Tobacco Development  
PROJECT LEADER: G. Gellatly  
PERIOD COVERED: July, 1987

#### I. UNFOLDED STRIP

- A. Objective:** To evaluate processing and structural characteristics of unfolded strip for improved filler length and cigarette quality.
- B. Results:** DBC bright strip was unfolded in the 8" tower at a rate of 500 lbs/hr at 490°F and 90 fps. An MF blend and a blend in which bright substituted burley was made of this product and normal DBC bright from the same lot. Filler from the unfolded strip and its control was also made in DIET.
- C. Plans:** To evaluate the performance of filler and DIET from unfolded strip in cigarettes and determine the potential of this project by September.

#### II. TMCI-ASTA SHEET

- A. Objective:** To develop a subjectively and physically acceptable reconstituted tobacco sheet using the TMCI process and PM-RCB technology for international application.
- B. Results:** The objective of this project was agreed upon by PM Management from Spain, TTG and R&D to be subjective parity of ASTA from Spanish feedstock with US-RCB at a 6% inclusion in a Spanish Marlboro.

Monique RCB was made using U.S. feedstock and sent to USA for subjective comparison with US-RCB made from the same feedstock. When modifications to the Monique plant to produce ASTA sheet are completed in late August, samples will be sent to USA for inclusion in the above comparison.

**C. Plans:**

1. To produce a physically and subjectively acceptable ASTA sheet from Spanish feedstock in FTR in the modified Monique pilot plant in September. Three of the best sheet candidates judged by subjective evaluation in FTR will be sent to USA for evaluation of blended cigarette subjectives and survivability.
2. Continue the development of Spanish ASTA in Tarragona when this plant is completed in November.

200117403

PROJECT NUMBER: 1806  
PROJECT TITLE: New Tobacco Processes  
PROJECT LEADER: S. R. Wagoner  
PERIOD COVERED: July, 1987

## I. CIGARETTE QUALITY

- A. Objective: Evaluate cigarette quality improvements using tobacco materials produced from new and/or less costly technologies.
- B. Results: Compacimetric testing was completed on the cigarettes made from superheated and control fillers, each produced with and without the Rothman's cross country unit prior to the cutter. The runs with the cross country infeed were not significantly different from those with the paddle feed, and both superheating trials produced a 0.3 mm firmness improvement versus nonsuperheating. Thus, the firmness benefit gained from superheating was not reduced by the cross country unit.

Also, the cigarettes were subjectively evaluated by the Domestic Cigarette Development Panel. Compared to the paddle feed/nonsuperheated control, the paddle feed/superheated and cross country/nonsuperheated combinations were found to be different, while the cross country/superheated trial was not different.

Since the Semiworks tunnel has been relocated to Louisville, a quotation was requested from Hauni for an HT tunnel to process 1000-2500 lb/hr of cut filler at 21% moisture. The price is DM 190,500 (approximately \$100,000) with a delivery of seven months.

- C. Plans: Currently, a summary report of program results is being written to support the purchase of the Hauni HT tunnel.

## II. STEM PROCESSING

- A. Objective: To provide a process for the direct conversion of stem materials into useable filler.
- B. Results:

Runs on the upgraded stem line have produced approximately 5000 lb of shredded, washed bright stem for future use by the ART facility. Individual rates of 1000, 350, and 550 lb/hr can be accomplished for the conditioning, shredding, and drying operations, respectively.

Check-out runs on the mono-basic potassium citrate application line have demonstrated the capability of preparing the solution from citric acid and either potassium hydroxide or tri-basic potassium citrate. An automatic titrator has been set up to determine solution concentration and casing level on the stems.

In addition, samples of the casing solution and cased stems were provided to Tobacco Microbiology (Project 1902) for evaluation of microbial growth.

Laboratory experiments were conducted washing bright and burley shredded stems with 60, 70, and 80°C water for various times to determine the effect on nicotine and hot water solubles removal. Chemical analysis is not yet available.

- C. Plans: After building the shredded stem stockpile to the required quantity, the new Sprout-Bauer refiner will be installed, which should increase the shredding rate to 1000 lb/hr. At that time, modifications will be made to the dryers to increase their capacity.

### III. BINDER DEVELOPMENT

- A. Objective: Develop methods to produce binder systems for the foamed bound rod, bonded ends, and reduced density rod programs.
- B. Results: Genu X7991 from Hercules foamed well at a 40% concentration in the laboratory. Foam density, molecular weight, and viscosity values were promising, and a larger batch was requested. Two samples from Grindsted, TSP-091 and 092, were also characterized in the laboratory and found to be acceptable. These were prepared at concentrations of 45 and 40%, respectively, for cigarette making trials.

Modified Smoking Materials (Project 1503) requested batches of in-house degraded citrus pectin for spray drying tests, ultrafiltration runs, and cigarette making trials. The ultrafiltration runs have shown the 100,000 molecular weight screen to be blinded by calcium aggregates of pectin. The effect of the aggregates on foam and cigarette quality will be evaluated, and could lead to the setting of a calcium specification for the vendors.

In support of the bonded ends program, polydextrose solutions were prepared. Also, solutions of 5% CMC, 1% xanthan gum, and 25% degraded citrus pectin were made for laboratory spotting tests.

- C. Plans: Continue to provide support as required for the above programs. Characterization of binder solutions produced with the continuous degradation process will require major effort in the short term.

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PROJECT NUMBER: 1101  
PROJECT TITLE: Entomological Research  
PROJECT LEADER: D. L. Faustini  
PERIOD COVERED: July, 1987

## I. CIGARETTE BEETLE (CB) PHYSIOLOGICAL STUDIES

- A. Objective: To conduct research investigations designed to produce results that lead to the control of the CB growth and development.
- B. Results: Methoprene and hydroprene treated liners did not appear to control the CB based upon examination of the tobacco contained within the liners. A substantial number of F1 generation emergence occurred 3 months after CB exposure.

The effects of the Dec. 1986 winter PH<sub>3</sub> fumigation have shown that CB emergence in 1987 was affected. Trap records showed 140 CB for 1987 and 104,000 CB for 1986 prior to the July fumigation period. This indicates that cold weather fumigation kills the overwintering larva and therefore reduces the biotic potential (e.g., subsequent generations).

Warehouse temperature studies indicate the CB emergence occurs when the commodity temperature reaches 70°F. Ambient temperature does not appear to differ from a hogshead at 16' or 4'; however, temperature differences do occur between hogsheads at these heights.

E. References:

S. Drew, Notebook No. 7850, pp. 190-194.

M. Minor, Notebook No. 8360, p. 51.

M. Minor. Comparison of trap counts during prefumigation period of 1986 and 1987. Memo to D. L. Faustini. July 21, 1987.

## II. SERVICE TO OTHERS

- A. Objective: To conduct and provide technical services to areas outside R&D.
- B. Results: One hundred percent CB mortality was achieved at the 20th Street tobacco conditioning chambers employed for Phytosanitary Certification of export tobacco.

Phosphine treatment of export cut filler in Tyvek® bags yielded 100% CB mortality of all life stages tested.

Assistance was provided to QA for the July fumigation at Leaf Storage facilities in Richmond, Cabarrus and Louisville. R&D monitored contractor activities and gathered data on wet deactivation method of phosphine residues.

A recommendation was made to QA-Louisville to eliminate the use of blacklight traps as the method to determine CB infestations. Infestations should be monitored using pheromone.

C. References:

Deubler, R. C. Memo to D. T. Wagner. Documentation of Beetle Kill at 19th and 20th Streets. July 13, 1987.

J. Machett. Memo to Distribution. MT in Boxes. July 22, 1987.

Faustini, D. L. Memo to D. W. Hardin. Blacklight Trap Use in the M/C Environment. July 22, 1987.

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PROJECT NUMBER: 1620  
PROJECT TITLE: Electrophysiological Studies  
PROJECT LEADER: F. P. Gullotta  
WRITTEN BY: I. Wannamaker  
PERIOD COVERED: July, 1987

NASAL EVENT-RELATED POTENTIALS (NERPs)

OVERALL OBJECTIVE: To develop methods by which to objectively and reliably evaluate human responses to cigarettes, smoke constituents and tobacco flavorants.

I. CONCENTRATION RESPONSE STUDY

- A. Objective: To further differentiate among olfactory, trigeminal and mixed stimuli by investigating the relationships among nasal event-related potential (NERP) measures, stimulus concentrations and subjective estimates of odor intensity.
- B. Results: Repeated measures analyses of variance have been completed for all compounds. Factors analyzed included main effects for concentration, hemisphere and electrode position as well as concentration x hemisphere, concentration x electrode position and concentration x hemisphere x electrode position interaction effects. Post hoc analyses (Duncan's Multiple Range Tests) are currently in progress on those factors yielding overall statistical significance in order to more closely examine the sources of the differences.
- C. Plans: Complete multiple comparisons and write Annual Report by the end of August.

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PROJECT NUMBER: 1702  
PROJECT TITLE: Aerosol Generation and Filtration  
PROJECT LEADER: K. A. Cox  
PERIOD COVERED: July, 1987

## I. OPTICAL PROCESSING

A. Objective: Develop optical processing methods for pack inspection

B. Results:

### Optical Processing Laboratory (C. Harward)

The renovation of D-109 for the optical processing laboratory is very near completion. Almost all of the equipment ordered for the laboratory has been received and moved into the lab. The imaging system to be used for the acquisition of digital images of cigarette packs has been set up and tested by the Computer Application Division personnel (R. Maher). Electronic problems have been encountered with the imaging board and a replacement requested from the vendor. Once an ethernet board is installed and tested, the system will be transferred to the optical lab.

### Evaluation of a Commercial System for Pack Inspection

Global Holonetics of Fairfield, Iowa has developed a system that uses optical processing techniques for on-line inspection problems. It was evaluated and found to show promise for the inspection of cigarette packs. A system will be obtained for further evaluation in manufacturing when it becomes available.

### Numerical Methods Development (K. Cox)

The Design of one or more of the components of an optical pack inspection system requires a number of 'training images' of acceptable and unacceptable packs as well as some numerical algorithm for determining the design specifications of the optical components from the training images. In particular, one very promising optical inspection system uses a synthetic discriminant function as a matched spatial filter in a correlator. The filter is a computer generated hologram (CGH) which must be designed so that the system output contains a peak of unit height for acceptable images but no peak for unacceptable images. A numerical algorithm for designing such a CGH has been developed. The new algorithm is expected to overcome some of the deficiencies of earlier approaches, allowing for larger training sets, high resolution images and small differences between acceptable and unacceptable images.

C. Plans: Establish a library of video images of both acceptable and unacceptable packs. Focus initially on three different pack types.

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## II. SIDESTREAM SMOKE MEASUREMENTS (C. Morgan)

- A. Objective: Carry out a systematic investigation of the effects of cigarette design parameters on sidestream visibility, particulate generation rate, and mean particle size.
- B. Results: We have found it informative to report a new quantity, the RPF (relative particulate fraction), in addition to the MBR (mass burn rate) and sidestream visibility. The RPF is defined to be the fraction of the mass released into the air that is the form of a visible aerosol. This number ranges from .012 to .058 and is a measure of the efficiency of the cigarette for converting its mass into a visible aerosol (a low efficiency is desirable). At constant aerosol particle size, the visibility (extinction coefficient at the Mercury green line) should be proportional to the product of MBR and RPF. The division is useful since it allows us to separate the more trivial reduction in visibility which results from a reduction in MBR from the more interesting reduction which results from a reduction in the RPF. Of the cigarettes we have examined, those made from wrappers containing Mg(OH)<sub>2</sub> exhibit the smallest RPF.

## III. SUBJECTIVE IMPACT OF ENHANCED VAPOR DELIVERY (D. Simpson)

- A. Objective: Design a series of cigarettes which, at a given puff position, have identical TPM delivery, RTD, dilution and mean aerosol particle size, but distinctly different vapor/particulate ratios. Evaluate for subjective differences.
- B. Results: A series of cigarettes (series A) with conventional CA filters (designed earlier) have been fabricated and submitted for RTD selection. A second series of cigarettes (series B) employing impactor filters have been designed and drawings made of the filter elements (Dick Cosner).
- C. Plans: Submit the series A cigarettes for puff x puff and vapor/particulate analysis in order to determine if the design targets have been achieved. Machine a small number of the impactor filters for series B and submit the cigarettes for similar analysis.

## IV. REMOTE SENSING OF FILLER DENSITY (T. Nguyen)

- A. Objective: Evaluate the potential of light transmission measurements for the remote sensing of the density of tobacco filler.
- B. Results: We have previously derived an equation describing the dependence of the extinction coefficient of a bed of tobacco on the density of the bed and the size of the shreds comprising it. The extinction coefficient of a 6 mm deep bed of tobacco was measured over a range of densities and found to be well described by the equation.

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- C. Plans: Repeat the extinction measurements for a bed of depth equal to that which would be encountered on a cigarette maker. Determine the accuracy with which a density measurement could be made as a function of the cross sectional area of the beam presented to the bed.

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PROJECT NUMBER: 1704  
PROJECT TITLE: Supercritical Fluid Processes  
PROJECT LEADER: J. L. Banyasz  
WRITTEN BY: T. M. Howell  
PERIOD COVERED: July, 1987

## I. LOW NICOTINE

- A. Objective: Provide product support for Flavor Development and for Cigarette Testing.
- B. Results: Bright, burley, and oriental tobacco components and two special strip blends were extracted individually at the standard Bremen IV control conditions. An extraction run using washed Japanese burley stems as scrubber was also made. The resulting extracted blends were given to Flavor Development for subjective evaluation. The bright, burley, and oriental components gave extraction efficiencies of 85%, 95%, and 76%, respectively. Burley extraction efficiency was reduced to 86% when run at 31% OV in lieu of the standard 25% OV.

An extraction of Full Flavor Blend, which included sheet material made from Bremen IV spent scrubber stems, resulted in a nicotine reduction of 77% as compared to 92% for a control run. Residual monopotassium citrate in the sheet material appears to inhibit nicotine extraction from the blend. The extracted blend has been forwarded to Cigarette Testing.

- C. Plans: Continue support as requested.

## II. LOW NICOTINE

- A. Objective: Optimize stem useage as an adsorber.
- B. Results: Increasing the OV of BL burley stems from 35% to 41% was observed to dramatically alter the nicotine profile throughout the adsorber bed. Stems at 41% OV gave a nicotine profile which was a significant shift from the bottom of the bed to the head of the scrubber. Normally, the bottom layer of stems contains ca. 1.2% nicotine. The higher OV stems bottom layer contained ca. 0.7% nicotine. In addition, a run using unwashed bright stem at 41% OV gave an extraction efficiency of 95%. Control 35% OV unwashed bright stems give only 85% reduction. This implies that raising the stem OV may reduce stem requirements. Three extraction runs using bright WFSS at 25%, 35%, and 45% OV in a single vessel scrubber design were made to further evaluate the nicotine profile throughout the scrubber bed. Results are pending.

The use of a dynamic OV profile in the stems was proposed and tried. A run using half the standard stem amount in a combination of 46% OV BL burley stems at the top and 23% OV BL burley stems at the bottom of the scrubber resulted in a 90% nicotine extraction

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efficiency. Further work will include how the high OV of the stems affects the tobacco OV during extraction.

C. Plans: This work is ongoing.

### III. LOW NICOTINE

A. Objective: Evaluation of alternate extraction conditions.

B. Results: A low pressure extraction run was made at 2400 psia and 62 °C. These conditions were chosen in order to be outside the region in which the spinodal line could be reached if an uncontrolled depressurization of CO<sub>2</sub> took place. A CO<sub>2</sub>-to-tobacco ratio of 250 : 1 was required to obtain 97% nicotine reduction. The tobacco was submitted for subjective evaluation.

C. Plans: No further work is planned.

### IV. LOW NICOTINE

A. Objective: Provide technical support for the ART Facility Group.

B. Results: Tobacco waxes were recovered from a Bremen IV let-down extract. The solid waxes were found to have a density of 0.92 g/cc and a wide melting range of from 54 °C to 80 °C. An excess amount of the waxes was placed in the microextractor system, and their solubility in liquid CO<sub>2</sub> was measured to be as follows:

<u>CONDITIONS</u>	<u>CONC. ppm</u>
11 °C @ 900 psi	421
11 °C @ 1500 psi	513
11 °C @ 3000 psi	850
19 °C @ 900 psi	514

This information was forwarded to the ART Facility Group for use in system design.

C. Plans: Work is ongoing as requested.

### V. LOW NICOTINE

A. Objective: Relocate the 7th floor lab to D201.

B. Results: Modifications to the D201 laboratory have begun and are expected to be complete in another month.

C. Plans: Design and fabricate a new one liter extractor system in D201 prior to shutting down the 7th floor lab and relocating.

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PROJECT NUMBER: 1706  
PROJECT TITLE: Thermal and Combustion Processes  
PROJECT LEADER: D. B. Losee  
PERIOD COVERED: July, 1987

I. PROJECT ART (B. Waymack)

- A. Objective: Determine the extent to which nicotine can be reduced by low temperature processing of tobacco.
- B. Results: J7MH filler has been treated at 70°C, 76°C, and 85°C for five hours in flowing helium. Preliminary results at 85°C have shown that the nicotine (determined as pyrolysis nicotine) can be reduced by approximately 20%, while a sample treated at 70°C is reduced by 10%.
- C. Plans: Investigate effects of water and  $\text{NH}_4\text{HCO}_3$  pretreatment on nicotine reduction. Establish other major components lost during this thermal processing.

II. LOW DENSITY ROD PROGRAM (B. Waymack, D. Driscoll)

- A. Objective: Characterize pectin binder degradation to establish if alternative treatments can lead to possible end product improvement.
- B. Results: Examination of degraded citrus pectin (provided by Sue Wrenn) and a control has shown, using EGA/MS, that the thermal degradation does not significantly alter the acid and ester functionalities. There are preliminary indications from DTG and EGA/MS that a high temperature (~750°C) ignition occurs in the control. This 750°C ignition is no longer present after degradation. The possible role that calcium plays in this phenomena is being explored.
- C. Plans: Continue pectin characterization with the aim toward providing alternative treatments of pectin to achieve the desired binding properties.

III. LOW DENSITY ROD PROGRAM (D. Driscoll)

- A. Objective: Establish utility of TGA as a quality assurance tool for pectin binders.
- B. Results: TGA comparisons of percent weight loss for specific temperature regions demonstrate excellent reproducibility within each batch of undegraded citrus pectin. The only statistically different weight loss region is between 30°C and 170°C for two batches of undegraded citrus pectin. These differences are due to either different drying or packaging conditions.

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Differences in the 130 - 350°C region appear sensitive to the extent to which pectin has been degraded. In addition, as noted above, it appears that degraded pectin ignites at a lower temperature than the control.

- C. Plans: Continue batch sampling of both undegraded and degraded samples of pectin using TGA.

#### IV. OPERATIONS SUPPORT (P. Henderson)

- A. Objective: Establish operating specifications for tipping adhesives.

C. Results: The flow characteristics of several tipping adhesives which are currently used in the Manufacturing Center have been evaluated at high shear rates. With the exception of one adhesive, all tipping adhesives examined thus far reach a critical shear rate above which they agglomerate. These critical shear rates appear to be characteristic for the various adhesives. Substantial variations in these critical shear rates are seen among adhesives which have the same nominal Brookfield viscosities.

Below these critical shear conditions, all of the adhesives undergo shear thinning. In this region a linear regression of shear stress versus shear rate data appears to provide a meaningful measure of operating viscosity. Each one of these best fit viscosities is substantially less than the corresponding Brookfield value.

- C. Plans: Explore the effects that small changes in temperature may have on these critical shear conditions.

#### VI. OPERATIONS SUPPORT (D. Driscoll)

- A. Objective: Support Quality Assurance and Operations Services in implementing thermogravimetric analysis as a screening test for incoming adhesives.
- B. Results: Diagnosed hardware and software problems on two newly acquired Perkin Elmer TGA-7 instruments. Trained personnel on the use of the TGA-7 after above problems were solved.
- C. Plans: Trouble shoot software, documenting programing errors, while writing the method to be implemented in QA for screening adhesives.

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PROJECT NUMBER: 1708  
PROJECT TITLE: Physical and Chemical Properties of Tobacco  
PROJECT LEADER: M. E. Counts  
PERIOD COVERED: July, 1987

#### I. ALTERNATE HUMECTANTS

- A. Objective: To find a plasticizer/humectant system that provides acceptable sheet materials without glycols.
- B. Status: In testing different ways for introducing propylparaben into BL Plant process, it was found that sheet subjectives were acceptable when propylparaben was added to the final slurry. Subjectives were not acceptable if propylparaben was added in the starting chemicals at the beginning of the process. Based on this, sheets of RCB were made in C Pilot Plant to test the two approaches for introducing K-propylparaben into RCB. These sheets have been made into cigarettes and are currently undergoing subjective analysis.

#### II. GLYCERIN FREE SHEET

- A. Objective: To provide acceptable RLTC, RL150B, and RCB sheets for the European market which are glycerin free.
- B. Status: Subjective and physical evaluation of glycerin free RLTC and RL150B sheets was completed. Results indicate that, if glycerin is replaced with an equivalent amount of PG, no significant differences in either subjectives or physical properties are detected when compared to respective controls containing standard amounts of glycerin and PG.

#### III. MECHANICAL PROPERTIES OF BONDED CIGARETTES

- A. Objective: To characterize the response of bonded and unbonded cigarettes to compression.
- B. Status: Force-displacement tests were done on Marlboro rods (from packs) and pectin-steam bonded 725 mg test cigarettes at a constant strain rate of 0.05 mm/second. The force-displacement curves for the cigarette types were different. The higher initial force vs displacement slope for the bonded cigarette was indicative of higher stiffness. The slope change decreased at higher loads and eventually was parallel with the force-displacement curve of the Marlboro control. The bonded cigarette slope deflection may signal bond breakage. This approach is encouraging and will be continued with suitable bonded rods and unbonded controls of the same density, with and without binder.

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#### IV. MECHANICAL PROPERTIES OF PACK OVERWRAP

- A. Objective: To compare stress relaxation properties of various pack overwraps.
- B. Status: A memo describing the differences between Marlboro and Winston overwraps was issued. Manufacturing personnel are being consulted, and future work plans will be designed according to the problems associated with films.

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PROJECT NUMBER: 1730  
PROJECT TITLE: Plant Cell & Tissue Culture Research  
PROJECT LEADER: I. L. Uydess  
PERIOD COVERED: July, 1987

## I. TOBACCO-IDENTICAL PRESERVATIVES

A. Objective: To develop procedures and to establish microbiological screens for the evaluation of new, tobacco-identical preservatives as replacements for and/or as adjuncts to propylparaben.

B. Status:

1. Research Proposal: A research proposal has been submitted to management outlining the methods and approaches that will be used to screen natural tobacco materials for preservative (antimicrobial) activity (1). The principal targets include:

- a. Readily available, known biochemicals that have been approved for use as preservatives on tobacco either in the US or Europe (such as those appearing on the GRAS, Hunter or other lists),
- b. Various chemical fractions that have been isolated from tobacco and that are currently available at PM R&D for further testing (such as duvatriene-diols, sucrose esters, etc., see reference 2),
- c. Aqueous and organic extracts of oriental tobaccos (see reference 1).

While some revisions are currently being made to this proposal, some of the preliminary experimental work has been initiated (see below).

2. Microbiological Screens: Several experiments have recently been conducted in a Nutrient Broth system containing 5% basal salts and MES buffer (pH 5.7) to test the potassium propylparaben screening procedures developed by Ola Mallory and Walter Hempfling. A pure culture of Bacillus coagulans isolated from chemically-altered ('spoiled') SEL served as the initial test organism and was grown in the presence of several concentrations of K-propylparaben (including 0, 100, 150 and 300 ppm). The growth of triplicate control and test cultures was monitored at 45 minute intervals by following the change in optical density at 650 nm over a period of 180 minutes (some cultures were followed overnight in order to obtain additional, end-point data).

3. General Microbiology: Procedures are being developed to store pure microbial cultures isolated from SEL as spores. This would replace the current practice of perpetuating such isolates over extended periods of time (months) via the

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intermittent passage and refrigerated storage of vegetative cells.

- C. Results: Repeatable results were obtained in each of five successive screening experiments corroborating the results reported earlier by Hempfling and Mallory (3). In each case, the 150 ppm level of K-pp produced about half-maximal growth while the 300 ppm level inhibited growth completely under the experimental conditions employed.

- D. Conclusions: None to be reported at this time.

E. Plans:

- C O
1. Test Materials: Discussions are currently being held with a variety of R & D personnel to help establish a list of prospective preservatives for testing. These materials would be selected from chemical fractions that have already been isolated from tobacco at PM R&D as well as from biochemicals which appear on published lists of known preservatives that have been accepted for use on tobacco either within the USA or internationally.
  2. Microbiological Screens: Additional test screens are planned for August in which 'sham' unknown preservatives (such as various antimicrobial agents, etc.) will be evaluated in blind tests against the propylparaben/B. coagulans control screen previously described.

F. References:

1. Uydess, I. L. and Teng, D. M., "Development of microbiological screens for identifying and evaluating tobacco-identical preservatives as replacements for propylparaben", PM Memo to Dr. E. B. Sanders, in press.
2. Personal communications, Dr. Richard Izac, Dr. Jeff Chan, and Dr. Rett Southwick.
3. Personal communications, Miss Ola Mallory and Dr. Walter Hempfling.

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PROJECT NUMBER: 1902  
PROJECT TITLE: Tobacco Microbiology  
PROJECT LEADER: D. J. Ayers  
WRITTEN BY: E. A. Crockett  
PERIOD COVERED: July, 1987

I. PROJECT ART BLEND SAMPLES

- A. Objective: To determine the potential for bacterial and fungal growth in the ART blend samples (OV = 23%) during storage at 108°F and to note any chemical changes that may occur.
- B. Results: MC Primary facility samples from the beginning, middle, and end of the silo dump were collected. In addition to a time zero sample, samples from these three collections were placed in containers and tested (1) after 4, 8, 12, 16, 24, and 48 hours of storage (108°F). Preliminary studies suggest that there was an increase in bacterial counts after 8 hours of storage in samples taken at the beginning and at the end of the dump; however, this increase was not observed in the middle of the dump samples.

The data for the mold/yeast samples are not available due to the longer incubation times needed to quantitate these organisms.

No chemical changes have been noted in selected samples from the preliminary study.

- C. Conclusions: None at this time.
- D. Plans: This work is ongoing.
- E. References:

1. Crockett, E. A., Special Report #86-055. Memo to J. Whidby; 1986 February 20.

II. MICROBIAL ANALYSES OF STRIP CASED WITH REGULAR BURLEY SPRAY AND RSB WITH THE ADDITION OF CLASS TOBACCO

- A. Objective: To microbially analyze Burley strip that was uncased (feed) and cased with either RBS (control) or CTB (test). These samples were obtained from a trial at the Louisville MC on 5/1/87.
- B. Results: After 12 weeks of storage there were no major differences in bacterial counts (1) between the control strip with the RBS vs the test strip with the CTB application. The control decreased by a factor of 4 and the test by a factor of 10. However, the feed exhibited an increase in bacteria by a factor of 10.

The mold counts (2) after 2 weeks of incubation increased from 2 to 35 (feed), 0 to 102 (control), and 3 to 168 mold colonies/g

(test). These counts, however, after the 12 week storage period, decreased to 7 or less mold colonies/g.

The yeasts (2) exhibited counts of 7 colonies/g or less after 12 weeks of storage.

C. Conclusions: After 12 weeks of storage, Burley strip sprayed with CTB (test) did not show any adverse microbial effects when compared to the strip cased with RBS (control). An increase in bacterial numbers was observed over the same time period for the Burley feedstock. The significance, if any, of the increase in the number of mold organisms in both the test and control strips after 2 weeks of storage has yet to be determined.

C.D. Plans: A memo detailing the results is in press.

E. References:

1. PM Notebook #8285, D. Turner; 8522, N. Thompson; 8181, D. Chadick; 8294, E. Crockett.
2. PM Notebook #8505, O. Mallory.

2001117421



PROJECT NUMBER: 1904  
PROJECT TITLE: Tobacco Physiology and Biochemistry  
PROJECT LEADER: H. Y. Nakatani  
PERIOD COVERED: July, 1987

#### I. LOW NICOTINE STUDY

- A. Objective: To investigate the biochemistry of the nicotine biosynthetic pathway at putrescine N-methyltransferase (PMT) and N-methylputrescine oxidase (MPO) and specifically to isolate PMT from tobacco root extracts.
- B. Status: A fourth group of hydroponically grown, Burley 21 (Bu21) tobacco plants (36 plants, approximately double the previous harvest) is being processed to obtain root extracts of a 40-65% ammonium sulfate fraction (2). A phenyl-sepharose column (hydrophobic interaction column) has been successfully used with tobacco root extracts, PM 27 and PM 28. It was discovered that the addition of 1.5 N NaCl directly to the 40-60% ammonium sulfate fraction was necessary in order to bind PMT to this column. This has eliminated the time and buffer consuming process of dialysis used previously. Four separate applications of tobacco extracts (at the ammonium sulfate stage) have been made to phenyl-sepharose columns and PMT-active fractions have been pooled and stored frozen at  $-80^{\circ}\text{C}$  (1,3).

A fraction from a phenyl-sepharose column was applied to a FPLC Superose 12 gel filtration column to obtain 0.05 ml fractions (see previous monthly). The PMT-activity was examined and an apparent molecular weight was estimated to be about 60 kD. The smaller sample volume did not increase the resolution, i.e., protein bands in the 40 - 60 kD molecular weight range were still present in the estimated 60 kD mw fraction. All fractions were examined by Phast system gels which were silver stained in order to visualize the proteins. New Aurodye and silver staining procedures were used successfully to detect the proteins (4).

A secondary antibody staining method using Auroprobe BL GAR, a goat antirabbit antibody, has been employed to examine PMT-active fractions. This new antibody procedure was utilized to examine PMT-active fractions eluted from a QAE-anion exchange cartridge (Cuno) and PMT-active fractions pooled and applied to a phenyl-sepharose column. Use of a silver enhancement technique along with the Auroprobe BL GAR allowed visualization of a positive cross-reaction of the phenylethanolamine methyltransferase (PNMT) antibody in conjunction with the goat antirabbit antibody to two proteins in the 55-60 kD molecular weight region (5).

Preparative gel electrophoresis was conducted on partially purified PMT-active fractions after the samples were dialyzed and freeze-dried. Two bands above 55 kD (based on the molecular weight of a glutamate dehydrogenase standard) were excised and electroeluted (6). These samples were subjected to dot-blot

antibody staining as described above and positive reactions against PNMT were observed for both fractions (5).

- C. Plans: The phenyl-sepharose column will be used to obtain initial purification of PMT from the ammonium sulfate fractions. Pooled fractions from phenyl-sepharose will be applied to the QAE anion exchange cartridge as the second step in the PMT-purification scheme. Preparative gel electrophoresis will be continued to obtain samples for future proposed antibody production.

D. References:

1. Malik, V. S. PM Notebook No. 8402.
2. Shelton, S. PM Notebook No. 8486.
3. Nakatani, H. Y. PM Notebook No. 8384.
4. Sherwood, K. R. PM Notebook No. 8416.
5. Mooz, E. D. PM Notebook No. 8296.
6. Sykes, A. PM Notebook No. 8526.

II. OTHER PROJECT ACTIVITIES:

Notification of the acceptance by TCRC of four abstracts submitted by members of Project 1904 for consideration has been received. Manuscripts are being revised and slides are being prepared for the presentations in October.

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PROJECT NUMBER: 6906  
PROJECT TITLE: Biological Effects of Smoke  
PROJECT LEADER: J. M. Penn  
WRITTEN BY: H. D. Garcia and W. R. McCoy  
PERIOD COVERED: July, 1987

#### I. INHIBITION OF EGF BINDING

- A. Objective: Establish the EGF assay and determine the effects of CSC in the assay.
- B. Results: Three replicate experiments were done to examine the effect of 2R1 CSC at 25 ug/ml on the binding of  $^{125}$ I-EGF at doses of 0.1 - 2.0 ng/ml to cultured 3T3 cells (1). The results showed that this concentration of CSC inhibited the binding of EGF by 20% compared to the DMSO solvent control while the positive control (1 ug catechol/ml) inhibited EGF binding by 50%. The DMSO solvent control (2%, v/v) did not inhibit EGF binding. These results are in excellent agreement with results from previous experiments (2).
- C. Plans: Evaluate several concentrations of CSC (e.g. 50, 75, 100 ug/ml) using increasing concentrations of  $^{125}$ I-EGF in an effort to obtain representative Scatchard and Lineweaver-Burk plots. This will allow determination of whether the observed inhibition is due to a reduction in the affinity of the binding sites for EGF or a reduction in the number of binding sites per cell or both.
- D. References:
1. Stagg, D. L. Notebook 8518, pp. 107-192.
  2. Stagg, D. L. Notebook 8518, pp. 67-106.

#### II. Diacylglycerol (DAG) Assay: Determination of the Maximum Tolerated Dose of 2R1 CSC for Cultured 3T3 Cells

- A. Objective: To establish whether treatment of 3T3 cells with high doses of CSC will cause detachment of cells from the culture dishes.
- B. Results: Treatments with CSC at concentrations up to 600 ug/ml (2 ml treatment volume) for treatment times of up to 2 hours in 100 mm glass Petri dishes containing  $2.8 \times 10^6$  to  $4.7 \times 10^6$  cells per dish did not induce any significant cell losses. The cell generation times, however, were longer than expected in this experiment.
- C. Plans: Examine factors affecting the growth rate (generation time) of 3T3 cell cultures (see below), then repeat this experiment at higher CSC/cell ratios.

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**D. References:**

Garcia, H. D. Notebook No. 8509, pp. 57-65.

**III. DAG Assay: Factors Affecting the Growth Rate of 3T3 Cells in 100 mm Petri Dishes**

**A. Objective:** Determine which culture parameter(s) is rate-limiting for growth of 3T3 cells so as to better define the cultures used in the experiments.

**B. Results:** The following parameters were examined: glass versus plastic dishes; 10 ml vs 20 ml of culture medium; 10% vs 20% calf serum in the medium; and cell inocula of 5E5, 1E6, 1.5E6 and 2E6 cells per dish. The cultures were all incubated for 3 days, then cell counts were obtained. These counts showed that: 1) these cells grow equally well in glass or plastic dishes; 2) the volume of culture medium is not limiting the rate of cell division under the experimental conditions used; 3) the generation times decrease (i.e., the rate of cell division increases) as the amount of serum per cell is increased.

**C. Conclusions:** After 3 days of incubation, these cultures appear to have depleted one or more necessary mitogenic factors from the serum, even at the highest serum/cell levels tested. This may prove useful for future experiments if it also means that the baseline level of DAG/cell is lower under these "growth factor depleted" conditions than for cells which are actively dividing.

**D. References:**

Garcia, H. D. Notebook No. 8509, pp. 66-76.

**IV. Acylarachidonyl Glycerol (AAG) in 3T3 Cells: TLC Method Development**

**A. Objective:** Establish conditions for thin layer chromatography of AAG.

**B. Results:** Two lipid standards, 1-oleoyl-2-acetyl-rac-glycerol (OAG) and 1,2-dioleoyl-rac-glycerol (DOG) were used to determine that 40 ug of sample per spot are needed to clearly visualize spots using iodine development. A solvent mixture of 7:3 toluene:ethyl acetate used to develop the TLCs was found to separate a mixture of these two closely related diacylglycerols.

**C. Plans:** Use the conditions established above in further experiments involving analysis of unlabelled lipids extracted from 3T3 cell cultures.

**D. References:**

Nixon, G. Notebook No. 7758, pp. 74-75.

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V. Protein Kinase C (PKC) Activity of 3T3 Cells: Electrophoretic Analysis of Protein Extracts from Whole Cells

- A. Objective: Determine the baseline electrophoretic pattern of total cell proteins from untreated 3T3 cells.
- B. Results: Cells were solubilized, subjected to gel electrophoresis and the gels stained. Good staining patterns for the cell proteins were obtained. Protein analyses were conducted on the cell extract; however, indications were that the high concentrations of SDS in the solubilization media interfered with the protein assay.
- C. Plans: Alternate methods of solubilizing the cells and accurately determining protein levels prior to electrophoresis are being investigated.
- D. References:

Tickle, M. H. Notebook No. 8515, Pp 105, 117, 129.

VI. PKC Activity in 3T3 Cells: Calibration of Autoradiograms

- A. Objective: Optimize the exposure times needed to obtain visible spots on XAR film with various amounts of  $^{32}\text{P}$ -ATP.
- B. Results: An exposure time of 24 hr at  $-70^{\circ}\text{C}$  allowed detection as low as 10 dpm of  $^{32}\text{P}$ -ATP.
- C. Plans: Additional experiments will be performed to investigate the reproducibility of these conditions.
- D. References:

Tickle, M. H. Notebook No. 8515, Pp 124, 144.

VII. Glutathione Depletion Assay (GDA): Reevaluation of Eight Model Cigarettes

- A. Objective: To recalculate rate constants (specific activities) using % GSH remaining versus weight of cigarette burned dynamically for the eight model cigarettes.
- B. Results: The results from eight model cigarettes were recalculated and evaluated. As the weight of filler burned per puff increased there was an increase in the aldehydes and CO produced. In addition, the more weight burned per puff resulted in less TPM and GP activity. These results suggest that more than just aldehydes are involved in GDA activity.
- C. Plans: To recalculate specific activities using % GSH remaining versus weight of cigarette burned dynamically and reevaluate the filter cigarettes designed to remove aldehydes.

D. References:

McCoy, W. R. Notebook No. 8484, pp. 160-163.

VIII. Salmonella/Microsome (S/M) Assay: Effect of Addition of Fructose to Burley CEL on IT CSC Activity

A. Objective: To investigate the effect of total reducing sugars (TRS) in filler on the S/M activity of the CSC.

B. Results: The addition of 6.75% fructose to Bu CEL on BrBW resulted in: (1) 18% decrease in activity with the addition of fructose to CEL (cooked prior to spraying); (2) a 16% decrease in activity with the addition of fructose to CEL (cooked before and after spraying); and (3) no decrease in activity when fructose was added to CEL (no cooking and/or just cooked after spraying). No statistical difference in activity could be demonstrated between control samples without fructose (cooked or uncooked).

C. Plans: To repeat the experiment with glucose and fructose addition levels of 15%.

D. References:

Thompson, L. H. Notebook No. 8516, pp. 97-98.

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PROJECT NUMBER: 6908  
PROJECT TITLE: Smoke Condensate Studies  
PROJECT LEADER: R. D. Kinser  
PERIOD COVERED: July, 1987

## I. TSNA PRECURSORS

- A. Objective: To determine the precursors of MS TSNA.
- B. Results: Significant levels of MS NAB were detected in the smoke from a low alkaloid burley filler with anabasine added at a target of 0.5%. A replicate addition of magnesium nitrate to "typical base web" confirmed the results reported last month: NNN and NAT increase dramatically, but no effect on MS NNK is observed. MS NO levels were found to be an order of magnitude greater in the smoke from the nitrate-treated tobacco.

Cyanuric acid was applied to burley tobacco as a possible NO scavenger. Addition of cyanuric acid at a target of 0.07% (moles filler cyanuric acid:moles MS NO = 1:3) resulted in no reduction in MS TSNA or NO deliveries.

Measurement by C. Morgan of dynamic and static burn rates for a control and a salt-treated cigarette demonstrated that both static and dynamic burn rates are reduced by the same amount for the treated cigarette. This indicates that the same amount of total filler was combusted dynamically for both control and treated cigarette, although puff counts were significantly greater for the treated cigarette.

- C. Plans: Replicate the anabasine addition experiment. Prepare a plan for future salt effects studies. Utilize a non-tobacco smoking material to determine optimum levels for cyanuric acid additions.
- D. References: Haut, S. A. Notebook No. 8468, p. 115.  
Morgan, W. R. Notebook No. 8506, p. 96.

## II. TSNA REMOVAL STUDIES

- A. Objective: To explore the possibility of extracting TSNA from stored tobacco using an extracting fluid compatible with current processing.
- B. Results: DBC burley was extracted in the apparatus fabricated by project personnel and described previously (Acc. No. 87-055, Project No. 6902 Monthly Progress Report). Solvents used were water, 95% ethanol, and 48.5% ethanol. Experiments were conducted at 1°C and 50°C. Differences in terms of TSNA removal were not as pronounced at 1°C, but at 50°C, the most selective solvent was 95% ethanol. Weight losses were only 14.5% and TSNA losses greater than 95%, compared to weight losses of ~25% and TSNA reductions of 75-80% for the other solvents.

C. Plans: Evaluate absolute ethanol, 2-propanol, 2-methyl-2-propanol, and hexane as solvents for TSNA removal.

D. References: Warfield, A. H. Notebook No. 8196, p. 170.  
Hansen, K. R. Notebook No. 8532, p. 1.

### III. ALTERNATIVES TO CARBON TRAPPING OF NICOTINE IN SCFE OF TOBACCO

A. Objective: To determine the efficacy of other nicotine trapping systems at the removal of TSNA from the process stream.

B. Results: Various burley stem samples were evaluated for TSNA content after being utilized as nicotine adsorbers in the R&D supercritical fluid extraction system. Best results in terms of TSNA levels were obtained with the extensively washed stems from the BL plant.

C. Plans: As requested, evaluate other nicotine adsorbers in terms of TSNA behavior.

D. References: Hansen, K. R. Notebook No. 8532, p. 1.

### IV. CARBONYL CONTENT OF SMOKE FROM A MODEL CIGARETTE OF LOW BIOLOGICAL ACTIVITY

A. Objective: To determine the chemical components responsible for the poor subjectives of smoke from a model cigarette of low biological activity (washed, shredded bright stems (WSBS)), and develop methods to decrease the levels of these components.

B. Results: Machine-made full flavor blend (FFB) cigarettes with 3 mg of THAM incorporated into a cellulose acetate (CA) filter exhibited a 68% reduction in formaldehyde delivered into mainstream, relative to a control FFB cigarette with normal CA filter.

C. Plans: Obtain subjective evaluation on the THAM-filtered FFB cigarettes and WSBS cigarettes with THAM on charcoal or THAM crystals in a plug/space/plug filter configuration.

D. References: Levins, R. J. Notebook No. 8413, p. 173.

### V. SUPPORT FUNCTION: CONDENSATE PREPARATION

A. Objective: To fabricate cigarettes, perform smokings, and prepare condensate as needed for biological and chemical analysis.

B. Results: Nine different cigarette fillers were prepared by spraying burley CEL on bright base web. The burley CEL was used as is, modified by addition of glucose or fructose, or hydrolyzed and subjected to ion exchange, pH adjustment, and glucose

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addition. Thirty smokings of 10 cigarette codes were performed to yield TPM or impaction trapped material for further analysis.

- D. References: Hellams, R. D. Notebook No. 8480, p. 146.  
Williams, D. L. Notebook No. 8530, p. 12.

2001117430

PROJECT NUMBER: 1720  
PROJECT TITLE: Analytical Microscopy  
PROJECT LEADER: C. E. Thomas  
WRITTEN BY: K. R. Sanders  
PERIOD COVERED: July, 1987

## I. STUDIES IN MICROSCOPY

- A. Objective: To obtain structural and chemical information on the morphology of low sidestream cigarette papers.
- B. Results: The above objective has been completed. Data and photographs collected from the papers were presented at the July Richmond meeting by B. Ferguson.
- C. Plans: A special report is in progress.
- D. References:  
Baliga, Vicki, PM Notebook #8412, p. 78.

## II. STUDIES IN IMAGE ANALYSIS

- A. Objective: Develop procedures using techniques of image processing and analysis for physical measurements of tobacco and related materials.
- B. Results: A method has been developed to measure the density of cocoa shell particles on the surface of burley tobacco after the application of burley spray. The method uses a 1-inch square of adhesive-backed paper to remove the particulates from the surface of a piece of Burley strip. The Intellect 100 image analyzer then quantitates the amount of particulates adhering to the paper.
- C. Plans: This method will be used to study the uniformity of the application of burley spray. The ability to collect a representative sample of the Burley strip after the spray has been applied will be the deciding factor in the usefulness of this method. For this reason the initial studies will concentrate on determining reproducibility on successive samples.
- D. Reference:  
Thomas, E., PM Notebook #8502, p. 37.

## III. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: Provide analytical support to R&D.
- B. Results:  
Scanning electron microscopy and energy dispersive x-ray spectroscopy were used to determine that a metal piece submitted

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by B. Laroy had an organic coating with small Ca crystals rather than a corrosive metal coating (1).

EDS was used to determine the elemental content of an insoluble material filtered from a solution of potassium triphosphate submitted by J. Allen. The insoluble material contained only the elements P and K (2).

Two Park 500 centrifuge residue samples submitted by J. Allen were examined for Ca and Mg phosphate crystals by SEM and EDS. All crystals analyzed which contained Ca also contained Mg (3).

Light microscopy was used to examine a broken ceramic garniture part submitted by W. Sanderson. It was determined that stress caused the part to break and that a small inclusion in the break was secondary to fracture (4).

At the request of S. Ganeriwala Marlboro and Winston polypropylene cigarette-package overwraps were examined by polarized light microscopy. Both were birefringent but the Winston overwrap had a slightly different orientation from the Marlboro overwrap (5).

A cigarette paper from an unknown sample was examined for surface structure and elemental composition by SEM and EDS at the request of R. Comes. The paper was perforated with a series of holes in parallel lines running the length of the cigarette. The holes were about 30-40 $\mu$  in diameter and appeared to be burned through the paper. The significant elements observed were Mg, K, and Ca with concentrations distributed as shown below.

<u>Element</u>	<u>Inside of paper, %</u>	<u>Outside of paper, %</u>
Mg	16	18
Ca	1	2
K	1	1

The above concentrations are very close to P6SU and P6SW, both low sidestream papers with 35 and 40% Mg(OH)<sub>2</sub> respectively, 5% CaCO<sub>3</sub>, and 4.5% K acetate. Close examination of the fibers showed a coating on the fibers similar to the Mg(OH)<sub>2</sub> coating found on the Mg(OH)<sub>2</sub> containing papers (6).

#### C. References:

1. Sanders, K., PM Notebook #8374, pp. 120-121.
2. Sanders, K., PM Notebook #8374, p. 118.
3. Sanders, K., PM Notebook #8374, pp. 124-129.
4. Sanders, K., PM Notebook #8374, p. 119.
5. Baliga, V., PM Notebook #8412, p. 77.
6. Baliga, V., PM Notebook #8412, pp. 77, 79.

2001117432

PROJECT NUMBER: 1740  
PROJECT TITLE: Flavor Components of Tobacco and Filler  
PROJECT LEADER: D. F. Ingraham  
PERIOD COVERED: July, 1987

## I. OVEN VOLATILES

- A. Objective: To identify the composition of oven volatiles.
- B. Results: Two samples each from five commercial blends were analyzed for compounds released during the OV experiment. Additionally, experiments were conducted to assess the effectiveness of the OV trapping apparatus.
- C. Conclusions: The trapping efficiency was slightly less for the blends than for other primary locations sampled to date.
- D. Plans: Four locations in Primary remain to be sampled to complete this study.
- E. References:  
PM Notebook #8469, pp. 88-92.

## II. RESPONSE TO ANALYTICAL REQUESTS

- A. Objective: Provide analytical support to R&D and Operations personnel and projects.
- B. Results: Analyses and investigations by the project personnel during the month of July included:

Nicotine analyses were performed on 135 samples from various submitters.

Four samples of Chinese menthol submitted by Ed Tucker were analyzed for purity by GC and optical rotation.

The remaining brands from a truck wreck in Maryland were analyzed for possible diesel and smoke contamination. Diesel contamination was observed on some materials while no smoke damage could be determined analytically.

Ethanol analyses were performed on seventeen samples for the Marlboro Standardization study. Ethanol values ranged from 30 to 209  $\mu\text{g}/\text{cigarette}$ .

Assistance in retrofitting a GC for capillary operation was provided to E. N. Maxey of Engineering.

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**C. References:**

1. PM Notebook #8340.
2. PM Notebook #8362.
3. PM Notebook #8471.

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PROJECT NUMBER: 1752  
PROJECT TITLE: Optical Spectroscopy of Tobacco and Smoke  
PROJECT LEADER: R. A. Fenner  
PERIOD COVERED: July, 1987

## I. MULTICOMPONENT SMOKE ANALYSIS

- A. Objective: To develop non-invasive analyses of multiple smoke components with simultaneous detection on a puff-by-puff basis.
- B. Results: XModem software has been installed on the Nicolet 160 SX FT-IR data system. This communications software will permit puff-by-puff data to be transferred from the Nicolet system to the VAX 8650. This new software is now being evaluated with the help of CAD.
- C. Plans: Continue with calibrations to permit quantitative puff-by-puff measurements.

## II. SUPPORT ACTIVITIES

- A. Objective: To provide analytical support to programs within R&D and for other departments as required.
- B. Results:
1. Tonka Bean Extract - Capillary GC/FT-IR was used to evaluate a tonka bean extract for Flavor Development. Major peaks identified were coumarin and derivatives thereof.
  2. Evaluation of HP's IRD - A trip was taken to Houston, TX to evaluate Hewlett Packard's GC/FT-IR detector. This dedicated detector system appeared to be exceptionally well designed and of better performance than obtained with our multipurpose Nicolet 60 SC GC/FT-IR system. Combined with HP's MSD, the IRD offers a powerful yet cost effective approach to GC/IR/MS.
- C. References:
- "Evaluation of Hewlett Packard's Dual IR/MS GC System," R. A. Fenner to Dr. R. Cox, July 15, 1987.

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PROJECT NUMBER: 1754  
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components  
PROJECT LEADER: J. B. Wooten  
PERIOD COVERED: July, 1987

I. SOLUTION NMR (R. Bassfield)

A. Objective: Determine the chemical structure of flavors and odorants.

B. Results:

1. The NMR chemical shift assignments of trimethyl isoxazole were made on the basis of model compounds and a two dimensional  $^1\text{H}$ - $^{13}\text{C}$  correlation spectrum. The assignments were confirmed by measuring the  $^{13}\text{C}$ - $^{13}\text{C}$  satellites in the 1-D  $^{13}\text{C}$  NMR spectrum.
2. Several two dimensional pulse sequences now exist that permit the assignment of carbon signals based on correlations of  $^1\text{H}$ - $^{13}\text{C}$  couplings over two or three bonds. These pulse sequences have been used with some success in the past, but technical problems have prevented us from using them routinely. Currently we are trying to work these problems out using 1-carvone as a model case. Potentially these pulse sequences are very valuable for making structural assignments in compounds having one or more hetero atoms or non-protonated carbons.

II. NMR SERVICE (J. Campbell and S. Marrs)

A. Objective: Provide rapid NMR service to PM scientists.

B. Results:

1. Steve Marrs has installed a new probe on the XL-300 that permits both a proton and carbon spectrum to be obtained without removing the sample from the magnet. This probe should improve sample throughput for service spectra since both the proton and carbon spectra can be obtained without the need to change probes and re-shimming the magnet. A "WALTZ" modulator that significantly improves proton-carbon decoupling efficiency and minimizes sample heating problems was also installed on the XL-300.
2. A new data field for CR numbers has been created in the NMR Database and NMR data from the CR file are being entered into the database by John Campbell. This field will permit the NMR spectra of the well characterized chemicals in the file to be quickly located. The data in the file are being used to cross check the chemical names and formulas that have already been entered into the database.

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PROJECT NUMBER: 1756  
PROJECT TITLE: Analytical Sensory Correlations  
PROJECT LEADER: B. W. Good  
PERIOD COVERED: July, 1987

#### INVESTIGATION OF ASHTRAY ODOR

##### A. Objective:

1. Define the "ashtray odor."
2. Establish a sampling protocol yielding extract with odor most similar to the "ashtray odor."

##### B. Results:

1. From limited subjective data from the cigarette butts of Marlboro s.p., the typical descriptors used for "ashtray odor" were stale, smoky, earthy, sooty, and stale tobacco smoke. The smoky note was very strong initially and decreased with time while the intensity of stale character changed in the opposite fashion. A steady state appeared to be established within 2 hours. Searching the Odor Profiling Database with the above descriptors produced a list of compounds consisting of pyridines, pyrazines, pyrroles, and guaiacol. Complete subjective data will be available from the Odor Profiling Panel.
2. Headspace sampling using a closed loop stripping technique with a charcoal trap and vacuum distillation were compared in the odor analysis of cigarette butts. Vacuum distillation yielded a yellowish aqueous solution with odor more similar to the "ashtray odor" than the headspace method. The principal difference of these two methods was in their ability to recover water-soluble substances which gave a strong smoky odor. Because of the hydrophobic nature of the charcoal, the headspace extract was more stale and less smoky.

C. Conclusions: Vacuum distillation of cigarette butts of Marlboro s.p. produced an extract most similar to the "ashtray odor."

D. Plans: To isolate and identify the substances responsible for the "ashtray odor" from vacuum distillation extracts by using odor sniffing and trapping of effluents from preparative GC and multidimensional capillary GC/MS.

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PROJECT NUMBER: 1757  
PROJECT TITLE: Analytical Flavor Specifications  
PROJECT LEADER: C. S. Kroustalis  
PERIOD COVERED: July, 1987

ANALYTICAL FLAVOR SPECIFICATIONS

- A. Objective: Develop analytical and subjective specifications for incoming flavors and materials used at the Flavor Center and appropriate QA facilities.
- B. Results: Final specifications were prepared for eight materials representing twenty-five vendors. Work was completed for about twenty other materials and specifications should be written by mid-August. Work is at various stages for the remaining twenty-five materials received to-date. We anticipate completion of analytical and sensory evaluations for these materials by the end of August and prepare specifications by mid-September.
- C. Plans: Complete analytical/sensory evaluations of the remaining materials on hand and begin evaluations of new materials on receipt.

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PROJECT NUMBER: 1758  
PROJECT TITLE: Tobacco Cell Wall Research  
PROJECT LEADER: G. H. Bokelman  
PERIOD COVERED: July, 1987

I. COMPOSITION OF DUST SAMPLES FROM THE CABARRUS PRIMARY (G. Bokelman and J. Stimler)

- A. Objective: Determine whether one or more components in the cigarette blend make disproportionately large contributions to the total amount of dust generated at the Primary in Cabarrus.
- B. Results: Using control blends, multiple linear regression analysis revealed that an accurate mathematical model for predicting the tobacco composition of these dust samples required only four predictor (or independent) variables: calcium, aspartic acid,  $\beta$ -methylvaleric acid (ratio of sample to standard) and cellulose index.
- C. Conclusions: It was found that there was a significant, but highly variable, content of sand in every dust sample. Comparison of the predicted compositions of the dust samples with the ratios of the cigarette blend components actually processed at the Cabarrus Primary led to a number of findings: (1) there is a relationship between collection site and composition of dust sample, (2) Oriental leaf appears to be the most friable of all the tobacco components, (3) bright lamina and burley lamina are the least friable tobacco components, and (4) stem, which is a major component of RL, has intermediate friability.

In summary, there were two major conclusions: (1) Oriental leaf is the only tobacco component which has a disproportionately great tendency to form dust and (2) sand is prevalent in all the dust samples.

- D. Plans: Analyses also will be run on another set of dust samples that has been requested from the Cabarrus Primary. In collaboration with Mary Ellen Counts, a friability study will be run on DBC bright, DBC burley and MT uncased filler to see if supporting data can be obtained regarding the tendency of Oriental leaf to form dust. In addition, silicon analyses will be performed on a number of tobacco components in an effort to determine the source of the sand in the Primary.

II. GEL PERMEATION CHROMATOGRAPHY OF DEGRADED CITRUS PECTIN (S. Baldwin)

- A. Objective: Determine the molecular weight distribution of one or more samples of degraded citrus pectin being considered for use as a foamed binder.
- B. Background: All methods for determining the molecular weights of polymers give averaged values. For example, the degraded citrus pectin samples presently being considered for use in the foamed

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binder program have molecular weights ranging from ~3,000 to ~5,000 daltons as determined by intrinsic viscosity. It is not known whether these samples are homogeneous or have polymodal distributions, the individual components of which might have different properties.

- C. **Results:** A variety of dextran molecular weight reference standards, some of which had to be custom ordered, have been obtained. Appropriate gels also have been ordered for the medium-pressure chromatographic system, which will be coupled to a refractive index detector.
- D. **Plans:** One or more of the most promising degraded citrus pectin samples will be fractionated by medium-pressure gel permeation chromatography. The resultant fractions first will be submitted for fast atom bombardment mass spectrometry to determine exact molecular weights. Subsequently, as time permits, these same fractions will be characterized by the methylation analysis procedures to determine monomeric linkage patterns. If more than one significant component is found in a degraded citrus pectin sample, an effort will be made to obtain sufficient quantities in order to make a preliminary evaluation of the physical properties of the respective fractions.

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PROJECT NUMBER: 2500  
PROJECT TITLE: Fundamental Chemistry  
PROJECT LEADER: J. I. Seeman  
PERIOD COVERED: July, 1987

I. FLAVOR/ODOR CHEMISTRY (Palen, Payne, Secor, Seeman)

A. Objective: To develop new technologies for smoke deliveries of desired flavorants; to prepare new substances for flavor/odor evaluation; to develop methodologies for the analysis of subjective data; to derive relationships between physicochemical parameters and subjectives.

C B. Results and Plans: We are in the process of preparing trimethylpyrazine 1 release agents. This chemistry is complicated by the fact that any procedure developed to date in related systems involves the condensation reaction of either a ketone (e.g., p-methoxyacetophenone) or an aldehyde (e.g., p-anisaldehyde 2) with the  $\alpha$ -C of an appropriate pyrazine. For 1, there are three such activated positions. We are currently examining purified materials prepared from 1 and benzaldehyde and the total, crude reaction product prepared from 1 and 2. Additional quantities of a tetramethylpyrazine:2,3-butanedione flavor release agent is being prepared. For mechanistic studies on the mechanism of pyrolysis of these compounds, two release agents of 2,6-di-t-butyl-4-methylpyridine (with benzaldehyde and acetophenone) were prepared.

Previously purified D-isomenthone was found to be undecomposed after 3 months at  $-20^{\circ}\text{C}$ . It was reduced to d-neoisomenthol in high yield and purity. Additional studies have now demonstrated that all four diastereomers of menthol crystallize well and can be purified as their terephthalates or 2,6-naphthalenedicarboxylates.

The structures of ca. 700 compounds that comprise the PM odor profiling study are now completely entered into the MACCS database. Corrections and added structures have been directly entered to the database. Names that were in error and typos have also been corrected. The chemical descriptors used in the Boelens odor database analyses have been converted into query language. Substructure will be conducted by a system of layered command programs. A series of fortran programs and command tiles have been prepared for the automated structure-odor search procedures. This sequence will be tested and evaluated in the coming month.

II. TOOL DEVELOPMENT IN MOLECULAR MODELING (Kao)

A. Objective: Develop the necessary algorithms and software to study physical properties of flavor and tobacco related molecules.

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- B. **Results:** Efforts continue extending the MOMM approach to TSNA's. Nitrogen-containing compounds are the initial target for this study. It is found that reexamination of the azoalkanes is a prerequisite to provide a consistent, universal, and unique force field for nitrogen-containing molecules. An improved force field is now completed for azoalkanes. New ab initio calculations have been performed to confirm and guide reparametrization. Both the structural and energetic results obtained from MOMM are superior to those previously reported. The average heat of formation deviation from experiment was 0.60 kcal/mol for 12 compounds, which represents an improvement of ca. 40% over the previous force field.
- C. **Plans:** The complete report of the improved force field for azoalkanes will be released shortly. The preliminary force field of C-nitroso compounds and organic nitrites is now ready to be finalized and extension of the developed MOMM parameters to N-nitrosamines will be carried out in due course. Hopefully, molecular properties (such as heats of formation, molecular structures, conformational energies, etc.) for TSNA's can be accurately predicted by this approach. Mechanistic studies of TSNA formation and degradation will be performed later.

### III. INFRARED IMAGING (Leister)

- A. **Objective:** To provide programming and data analysis support for the infrared imaging program.
- B. **Results:** Preliminary studies on image processor were completed. The goal was to locate the problem with 1000°C calibration point of blackbody source. A second study is underway. A FORTRAN program "WTAVE.F" was developed to calculate weighted averages for pixel luminances of calibration.

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PROJECT NUMBER: 2507  
PROJECT TITLE: Radiochemical Investigations  
PROJECT LEADER: S. B. Hassam  
PERIOD COVERED: July, 1987

## I. Mg TRANSFER

- A. Objective: To collect and analyze smoke for Mg levels.
- B. Results: Filtered cigarettes with wrappers containing 0, 12 and 35%  $\text{Mg}(\text{OH})_2$  (5 of each type) were smoked on a total impaction trapping smoking machine. The collected smoke condensates and butts, together with unsmoked cigarettes, were submitted for measurement of Mg by atomic absorption. A comparison of the Mg recovered in smoke vs. that in unsmoked cigarettes showed 99.6% recovery for the control (#3650, 0%  $\text{Mg}(\text{OH})_2$ ), 97.3% for #3652 (12%  $\text{Mg}(\text{OH})_2$ ), and 76.5% for #3654 (35%  $\text{Mg}(\text{OH})_2$ ). The reasons for the lower than expected recovery for #3654 are unclear. Based on total recovered Mg levels in smoke, the % transfer of Mg into sidestream condensate was 0.02% for control, 0.02% for #3652 and 0.04% for #3654. Greater differences were observed for Mg levels in the sidestream material that collects in the dome covering the cigarettes: 0.04% for control, 0.07% for #3652 and 0.22% for #3654.
- C. Plans: Continue investigation of low sidestream delivery cigarettes. Measure Mg levels by neutron activation.
- D. References: N.B. 8386; R. H. Newman, memo to R. N. Ferguson, "Mg Measurement in Low Sidestream Delivery Cigarettes", July 20, 1987.

## II. GAS PHASE ANALYSIS

- A. Objective: To develop collection and analysis procedures for sidestream gas phase analysis.
- B. Results: Gas chromatography analysis of  $\text{CO}/\text{CO}_2$  ratios in sidestream and mainstream gas phases of unfiltered cigarettes with and without  $\text{Mg}(\text{OH})_2$ -added papers was continued. Five cigarettes of each type from packs #3657, 3659 and 3661 were smoked. A summary of the instrumentation, smoking experiments and results is being prepared.
- C. Plans: Continue analysis of low sidestream delivery cigarettes. Continue development and improvement of the current method of analysis.
- D. References: N.B. 8154

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### III. INFRARED CAMERA

- A. Objective: Measure temperature distributions in cigarette coals and develop computer programs for data analysis.
- B. Results: A program to extract temperature vs. area information from the infrared camera images has been completed. Using this program infrared red images of cigarettes with  $\text{Mg}(\text{OH})_2$ -added wrappers were analyzed. A large variation in the temperature vs. area was observed for (1) the same cigarette at different times and (2) different cigarettes at the same charline position. These variations may reflect dynamic processes occurring during smoulder which is usually thought of as a steady state process with little temporal diversity.
- C. Plans: Complete calibration programs. Increase the number of smoking runs to improve sample comparisons. Develop techniques to investigate further dynamic aspects of the smoulder process.
- D. References: N.B. 8481; D. Leister, memo to D. McRae, June 4, 1987; D. Leister, memo to D. McRae, June 11, 1987; D. McRae and D. Leister, memo to R. Ferguson, June 15, 1987; D. Leister, memo to D. McRae, June 29, 1987.

### IV. SMOKE CHEMISTRY STUDIES

- A. Objective:
1. Compare total smoke distribution from  $^{14}\text{C}$ -octatriacontane-labeled cigarettes with and without  $\text{Mg}(\text{OH})_2$ -added wrappers.
  2. Analyze smoke components from  $^{14}\text{C}$ -octatriacontane-labeled cigarettes by high performance liquid radiochromatography (HPLRC).
  3. Synthesize  $^{14}\text{C}$ -labeled n-henhexacontane,  $\text{n-C}_{61}\text{H}_{124}$ , for use as a model compound in smoke transfer studies.
- B. Results:
1. Data analysis is in progress.
  2. HPLRC of  $^{14}\text{C}$ -labeled smoke components has been completed and data analysis is in progress.
  3. The key step of a proposed synthetic route was performed using unlabeled materials. Specifically tosylmethyl isocyanide was alkylated with 1-iodotriacontane. The resulting product was analyzed by  $^1\text{H}$  NMR, FTIR and EI/MS and concluded to be the desired intermediate 31-isocyano-31-tosyl-henhexacontane. Hydrolysis of this product gave the ketone 31-henhexacontanone, the precursor to  $\text{n-C}_{61}\text{H}_{124}$ .

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**C. Plans:**

1. Continue smoke distribution studies of radiolabeled cigarettes.
2. Perform radiochromatography analyses as needed.
3. Continue investigation of the synthetic route.

**D. References:** N.B. 8386; N.B. 8487; memo from S. B. Hassam to R. N. Ferguson, "Synthesis of Radiolabeled n-henhexacontane,  $C_{61}H_{124}$ ", July 3, 1987.

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PROJECT NUMBER: 2525  
PROJECT TITLE: Tobacco Chemistry  
PROJECT LEADER: S. Tafur  
PERIOD COVERED: July, 1987

#### I. NATURAL PRODUCTS ISOLATION

- A. Objective: To isolate and identify natural products with major emphasis on tobacco and tobacco products.
- B. Results: A Waters photodiode array detector was ordered, received and installed to upgrade our LC capabilities, especially for natural products isolation efforts and analytical LC studies.
- C. Plans: To integrate the new capability into current HPLC work.
- D. References: Core, M. Notebook No. 8417, p.116.

#### II. PROPYL PARABEN

- A. Objective: To find a suitable solvent as a replacement of PG for addition of propyl paraben to the RL process.
- B. Results: An HPLC procedure has been developed for the analysis of paraben methyl or propyl esters and the free acid, p-hydroxybenzoic acid. Using this method a study of the hydrolysis of 10% solutions of propyl paraben in various concentrations (3.4 to 7.4N) of ammonium hydroxide has begun.
- C. Plans: To determine the minimum concentration of ammonium hydroxide necessary to prepare a 10% propyl paraben solution which will last a week without any appreciable hydrolysis.
- D. References: Izac, R. Notebook No. 8379, p.122.

#### III. GREENHOUSE STUDIES

- A. Objective: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.
- B. Results: A fourth group of Burley 21 plants grown in hydroponic culture has been harvested for Project 1904 in support of the low nicotine program. The 36 plants produced about 12.4 Kg of fresh root tissue (346g/plant). A fifth group of 36 plants is in progress.

The  $^{14}\text{C}$  plant growth chamber has been refurbished with the assistance of A. Burton. A chamber run has begun to produce additional  $^{14}\text{C}$ -labeled bright and burley tobaccos for research purposes.

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The greenhouse phase of the culture x topping study has been completed. All leaves have been oven-cured and will be submitted for chemical analyses.

C. Plans: Continue to provide materials as requested and complete research studies in progress.

D. References: Bass, R. Notebook No. 8495, pp.50-54.

#### IV. SMOKE DISTRIBUTION OF FLAVORS

A. Objective: 1) To demonstrate the differences in smoke distribution for volatile flavors vs. flavor release compounds.  
2) To examine factors which may influence flavor distribution.

B. Results: Comparisons of smokestream distribution of anethole and menthol from menthyl anisyl carbonate (CR-2273) in various cigarette configurations have continued (1). While SS/MS ratios of the flavors from the release compound were similar for standard (KC 137-1) and 12% Mg(OH)<sub>2</sub> papers, the SS/MS ratio decreased in a 40% Mg(OH)<sub>2</sub> model due to both increased MS delivery and lowered SS delivery. The same observation was made for delivery of free anethole in a 40% Mg(OH)<sub>2</sub> model.

As shown previously, paper application of CR2273 along the sideseam of a normal paper leads to very low MS flavor delivery. Since the two sides of Mg(OH)<sub>2</sub> papers are quite different, the effect of applying CR-2273 to the inside vs. the outside of the paper was compared. In both 12% and 40% Mg(OH)<sub>2</sub> models, very low MS flavor deliveries were found by applying CR-2273 to the paper, but with no clear distinction between the sides of the paper. Application of CR-2273 to these papers, however, resulted in a decrease in overall recovery of released flavors. In addition, it was noted that with the Mg(OH)<sub>2</sub> papers the distribution of flavors in SS appeared to shift more to gas phase since a larger amount of flavor collected passed through a Cambridge pad into a back-up cold trap.

An increase in MS delivery of flavors from CR-2273 was shown when used with cigarettes having polypropylene filters. In another attempt to enhance MS flavor delivery from CR-2273, a high loading (20mg/cigt) of the release compound was placed at the front end (1.5cm) of Merit Menthol cigarettes. Neither the first three puffs nor the total MS delivery of anethole or menthol increased (2).

While fairly high levels of CR-2273 have not delivered distinct flavor to MS, a definitive SS anethole odor has been noted as expected.

An attempt to develop an ion chromatographic method for acetic acid in smoke was discontinued due to major interferences in TPM

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extracts. Method development has now been focused on a GC method which directly analyzes a TPM extract without acid derivatization. Initial analyses have indicated much lower MS levels than expected while SS levels appear normal (3).

C. Plans: Continue to examine flavor distributions upon smoking.

D. References:

1. Tafur, S. Notebook No. 8490, pp.72-73.
2. Tafur, S.; Grubbs, H.; Levins, R. SS/MS flavor distribution studies: Effect of heavy loading of anisyl menthyl carbonate (CR-2273) on the front end of Merit Menthol. Memo to R. N. Ferguson. 1987 July 20.
3. Keene, C. Notebook No. 8236, p.195.

V. SUPPORT ACTIVITIES

A. Objective: To provide requested assistance for Chemical Research activities.

B. Results: Assistance was provided to Lee Labs in Petersburg on the large scale preparation of poly isopropenyl acetate (1).

Ten handmade cigarettes each in four different cigarette papers were prepared using a blended filler sprayed with  $^{14}\text{C}$ -nicotine (2).

C. References:

1. Izac, R. Notebook No. 8379, p.122.
2. Bass, R. Notebook No. 8495, pp.50-54.

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PROJECT NUMBER: 6505  
PROJECT TITLE: Special Investigations and Methods Development  
PROJECT LEADER: D. C. Watson  
PERIOD COVERED: July, 1987

## I. X-RAY FLUORESCENCE (XRF)

- A. Objective: To provide qualitative and quantitative elemental data on tobacco, cigarette paper and materials evaluation samples.

C B. Results:

Qualitative Analysis

XRF data were used to define the elemental composition of seven paper samples. Inclusive were Mg, Al, Si, P, S, K, Ca, Fe, and Ti.

Analysis of degraded pectin samples continue to show Ca and K to be the major components.

Instrumentation

Problems with the Tracor 4020 sample tray have been corrected. The Tracor 5000 was received and installed. A faulty component in the power supply should be replaced by the first of August, at which time the 5000 will be fully operational.

C. References:

PM Notebook #8416, pp. 82-83.

## II. ATOMIC ABSORPTION

- A. Objective: To provide elemental determinations on tobacco and cigarette paper samples.

- B. Results: Magnesium - Two samples of cigarettes were analyzed for Mg content of the filler and paper. One sample was subsequently analyzed for Mg content of the mainstream and sidestream condensate, a sidestream wash, and cigarette ash plus butts. The data showed:

	Sample Code	
	#3652	#3654
Magnesium ( Wt. Percent)		
Filler*	0.48	0.40
Paper*	2.4	12.4
Magnesium (µg/cigarette)		
Mainstream	-	0.60
Sidestream	-	4.4
Sidestream Wash	-	27.2

Ash plus Butts

12100

\*Average of duplicates

Eight cigarette samples were analyzed for Mg content of the paper using both the routine extraction and the microwave digestion procedure. No differences were found.

C. References:

1. PM Notebook #8416, pp. 82-83.
2. PM Notebook #8394, p. 21.

III. PESTICIDE LABORATORY

- A. Objective: To provide analytical data for pesticides in offshore leaf purchase.

B. Results:

Three offshore samples have been analyzed for pesticides.

Evaluation of the herbicide derivatization and final cleanup steps have shown that the major source of error is the loss of the methyl ester of the herbicide and internal standard during the N<sub>2</sub> evaporation steps. Unfortunately the compound most affected is the internal standard. Steps have been taken to minimize the loss. Data collected does not demonstrate an obvious benefit to incorporation of the internal standard. Further review is in progress. Recoveries are quantitative for the herbicides and internal standard from a spiked sample.

Twenty-four samples were run for determination of FDC Blue #1 dye to assess its utility as a tracking agent in the foam binder utilization studies.

C. References:

PM Notebook #8499.

IV. PROJECT ART

- A. Objective: Determine the relative efficiencies of extraction of nicotine from stem material using various aqueous salt solutions.
- B. Results: Samples of bright and burley stem were extracted with 1% aqueous salt solutions with portions taken with time to determine the rate of extraction of nicotine from the stem.
- C. Conclusions: Comparisons of the aqueous solvent systems and tobacco stem matrix on the extraction rates of nicotine showed:

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1. Deionized water is not significantly lower in efficiency than 1% citric acid, potassium carbonate, or diammonium or monoammonium phosphate solutions.
2. Shredding the stem significantly increases the efficiency of nicotine extraction.

All extractions were done at room temperature with 5 grams of dry stem in 100 mL of solution.

- D. Plans: Samples of stem from various stem washings and from evaluations of new washing protocols will be analyzed to determine efficiency of the processes specifically as applied to Project ART needs.

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PROJECT NUMBER: 8101  
PROJECT TITLE: Cigarette Testing Services Division  
SECTION LEADER: Rebecca W. Kanipe  
PERIOD COVERED: July, 1987

## I. MARKET ACTIVITY

A. Objective: To monitor the introduction of new brands and brand modifications to existing brands and to report the domestic market activity on a monthly basis.

B. Results:

1. New Brands

Philip Morris is distributing Cambridge Full Flavor 85 and 100 cigarettes nationally. These cigarettes deliver 16 mg tar and 1 mg nicotine and will compete against Doral cigarettes (RJR).

R. J. Reynolds is distributing Camel Filter 100 cigarettes nationally. These cigarettes deliver 18 mg tar and 1.1 mg nicotine. This brand has the same packaging as Camel Filter 85.

Safer Smokes, Inc. of Fort Lee, New Jersey is distributing Bravo 85 cigarettes. The filler of this brand is 100% lactuca (a lettuce indigenous to Argentina) and contains no nicotine. These cigarettes sell for \$18.75 per carton.

2. Brand Modifications

Brown & Williamson has changed the filter on Kool Ultra 85 and 100 cigarettes back to the Actron (Barclay-type) filter. The filter on these cigarettes was changed to a conventional cellulose acetate filter in November, 1986. Barclay Menthol 85 and 100 cigarettes have been discontinued by Brown & Williamson.

Lorillard has increased the tipping paper length on Satin 100 (plain and menthol) and True 100 (32 to 34 mm). The tipping paper length on Kent Golden Lights 100 and Kent III 100 cigarettes has decreased (35 to 34 mm). Lower smoke deliveries were noted on the Kent III cigarettes as a result of increased ventilation.

## II. JAPAN MARKET

A. Objective: To evaluate Philip Morris and competitive cigarette brands on the Japanese market and to report the analytical results.

B. Status: The first Japan C.I. Report has been completed and published. This report includes analytical data and sales figures on thirty-one cigarette brands from the Japanese market.

### III. ANALYTICAL METHODS DEVELOPMENT AND SUPPORT

A. Objective: To evaluate and recommend analytical methods and new technology in support of programs for R&D and Manufacturing.

B. Status/Results:

1. Menthol Extraction Study: An extraction study has shown that a one hour, rotating extraction is sufficient for extracting menthol from TPM pads in 95% ethanol without impacting the precision of the method. The current standard procedure for menthol in smoke, Method No. S-8A, requires a two hour rotating extraction. The shorter extraction time will decrease the total analysis time by one hour. In addition, the decreased analysis time will increase laboratory efficiency in analyzing priority menthol samples.

2. Menthol on Foils using the InfraAnalyzer: The InfraAnalyzer procedure for the rapid determination of menthol on foil was presented to Diane Coleman from Louisville QA on July 2. The menthol on foil analysis is currently being performed in the QA laboratory using the gas chromatographic procedure. Bill Speicher has requested to have an InfraAnalyzer 450 set up in the QA laboratory for process control of mentholated foils. The four filters and wavelength coefficients for the analysis have been installed on an InfraAnalyzer 450. This instrument will be installed in Louisville on August 10 by CTSD personnel.

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